ENGINEERING OPERATIONS REPORT

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DESIGN, FABRICATION AND NONDESTRUCTIVE TESTING

OF

SIX EXPERIMENTAL AGCARB/INTREMOLD III CYLINDER ASSEMBLIES

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APRIL 1972

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ENGINEERING OPERATIONS REPORT

Design, Fabrication and Nondestructive Testing $of \\ Six \ \ Experimental \ \ AGCarb/Intremold \ \ III \ \ Cylinder \ \ Assemblies$

Project 143

April 1972

E. F. Thacher

Nozzle Extension Section

Approved:

C. M. Kawashige, Supervisor Nozzle Extension Section

L. A. Shurley, Manager

Nozzle, Pressure Vessel and Nozzle Extension Department

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PART C

INTREMOLD III CYLINDERS NONDESTRUCTIVE TESTING

I. INTRODUCTION

The nozzle assembly of the NERVA engine consists of a stainless steel convectively-cooled nozzle extending from the reactor interface to area ratio 24:1. At area ratio 24:1, the nozzle is joined to a fibrous graphite Nozzle Extension (NE). The NE is 4-1/2 feet in diameter at area ratio 24:1, 12 feet long and 10 feet in the diameter at area ratio 100:1, the exit.

AGCarb-101, a laminated graphite composite was selected as the prime candidate material for construction of the extension. This material, although well suited for the nozzle extension shell, is structurally deficient in cross-ply and interlaminar shear directions in the nozzle extension/nozzle flange (the only highly stressed region).

To circumvent these deficiencies, Intremold III, a graphite composite with potentially superior cross-ply and interlaminar shear strength properties due to graphite fibers in the block (interlaminar) direction as well as in the warp and fill has been selected as an alternate material. The manufacturing process is considerably more time consuming and therefore more expensive than for AGCarb. Therefore the application of Intremold III was to be limited to the flange area while the nozzle extension shell would be of AGCarb. In view of the fact that very little was known about the Intremold III material properties, the process itself, and means of joining Intremold III to AGCarb, an experimental subscale program was undertaken with the following objectives:

- 1. Demonstrate the fabricability of Intremold III subscale flanges
- 2. Develop an integral transition/joining method of Intremold III to AGCarb
- 3. Define the manufacturing process
- 4. Identify best suited quality inspection method
- 5. Evaluate structural properties of the product in the fabricated configuration.

The scope of the effort covered in this report was limited to (1) development of fabrication techniques for Intremold III flange and a transition to the AGCarb shell (through the first cure shown in Part A); (2) carbonization, densification and graphitization processes shown in Part B; and (3) evaluation of nondestructive testing methods, shown in Part C.

Termination of the NERVA program precluded the execution of the Intremold III material properties evaluation. Thus this phase of the originally planned effort was not conducted.

II. SUMMARY AND CONCLUSIONS

A. SUMMARY

To meet the objectives outlined in the Introduction, six subscale Intremold cylinder assemblies with a total of twelve (12) different concepts for transition to AGCarb, one on each cylinder end, were fabricated. Three of the cylinder assemblies were made by helically winding the hoop fibers and three were of orthogonal configuration (see Figures 1 and 2). The fabrication process used is summarized in the next section. Details of each manufacturing process are given in Parts A, B and C. The cross-sections of the cylinder wall along with the joint configurations fabricated or attempted are given in Figure 3.

The experience gained in cylinder fabrication identified process deficiencies and manufacturing flaws which should be avoided in the future. At the same time it was possible to define processes and manufacturing procedures that should be adopted for fabrication of the full size nozzle extension.

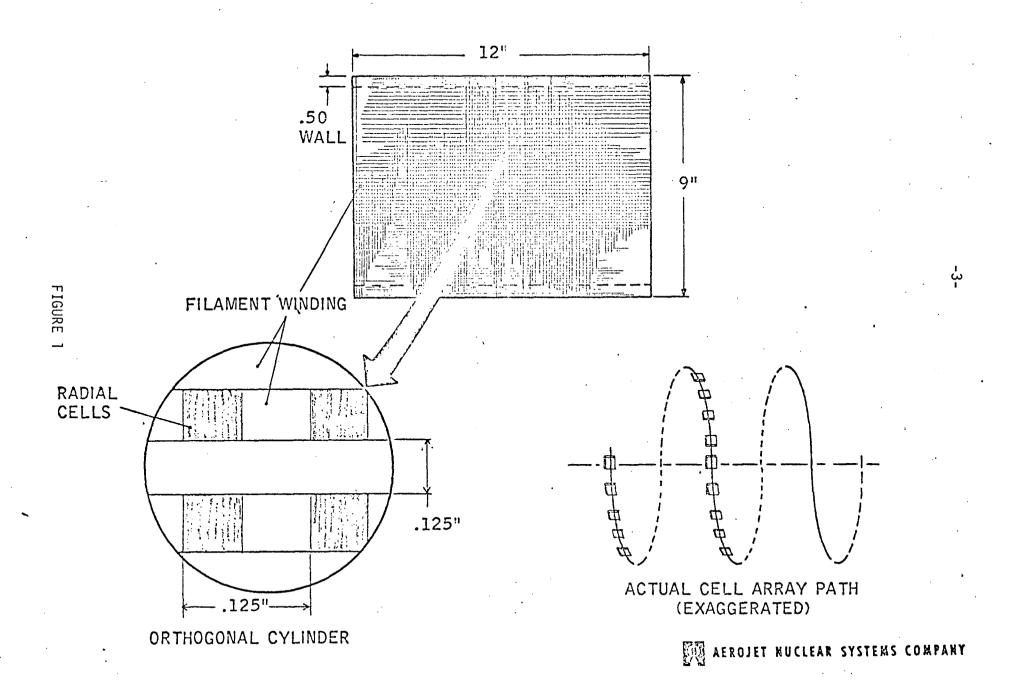
The objectives of the program were to: (1) demonstrate the fabricability of the Intremold III subscale flanges, (2) produce an integral transition from Intremold III to AGCarb material, (3) define a workable manufacturing process and (4) identify a best suited inspection method.

These objectives were met and the results are described in this report.

B. CONCLUSIONS

- 1. Either helical or orthogonal, Intremold III composite, full size nozzle extension flanges can be fabricated, based upon provision of appropriate tooling.
- 2. Without structural test evaluation no conclusion can be made regarding the structural superiority of the helical versus orthogonal configuration. The latter, however is easier and less expensive to fabricate.

METHOD OF CONSTRUCTION INTREMOLD III ORTHOGONAL



HELICAL CYLINDER

3. Of the twelve transition joints of Intremold III and AGCarb materials as shown in Figure 3, the Helical Assembly No. 1, Model B (Combtooth) and Orthogonal Assembly No. 3, Model A (Interleave) were found to be unfabricable.

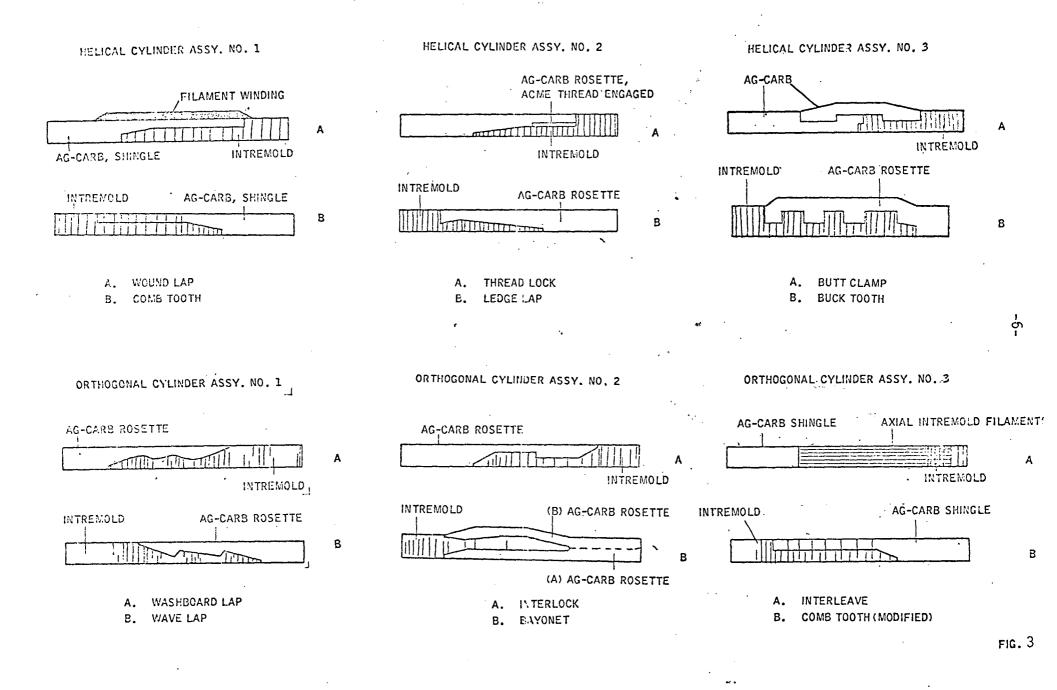
Helical Assembly No. 2, Model B (Ledge Lap) and the Orthogonal Assembly No. 3, Model B (Combtooth) were difficult to fabricate, and therefore not recommended for the future full scale use.

Orthogonal Assembly No. 2, Model A (Interlock) appears to be the most satisfactory and promising based on the fabrication experience defined in paragraph 4.

The remaining configurations produced are fabricable and may be suitable for full scale production, but with a varying degree of fabricability. The ultimate selection should be made only after structural evaluation data becomes available.

- 4. The optimum point in the sequence to fabricate the transition joint is after Intremold III cure. This eliminates damage to Intremold III from joint fabrication techniques.
 - 5. The carbonization/graphitization process revealed that:
- a. The cylinder assembly dimensional changes (shrinkage, generally) are less than 1%, but may require consideration in fabrication of the full size part.
- b. Three densification cycles, while fully adequate to raise the density of the AGCarb portion to 1.4 or greater, are insufficient for the Intremold III portion of the assembly. The Intremold portion was found to be not only less dense but also permeable as determined by alcohol penetrant tests. At the time of NERVA program termination it has not been determined how many more densification cycles would be required to eliminate the detected permeability.

INTREMOLD III TO AC-CARB TRANSITIONS



6. Nondestructive testing led to the conclusion that X-ray radiography and alcohol penetration were the only techniques found suitable for qualitative evaluation of the produced parts. Even though the radiography revealed a great amount of structure detail, a quantitative inspection can only be performed after development of meaningful standards.

III. CYLINDRICAL ASSEMBLY MANUFACTURING PROCESS

Figure 4 illustrates the process adopted after the version initially used had proven unsatisfactory. The temperatures and pressures shown are the maximums reached during each step.

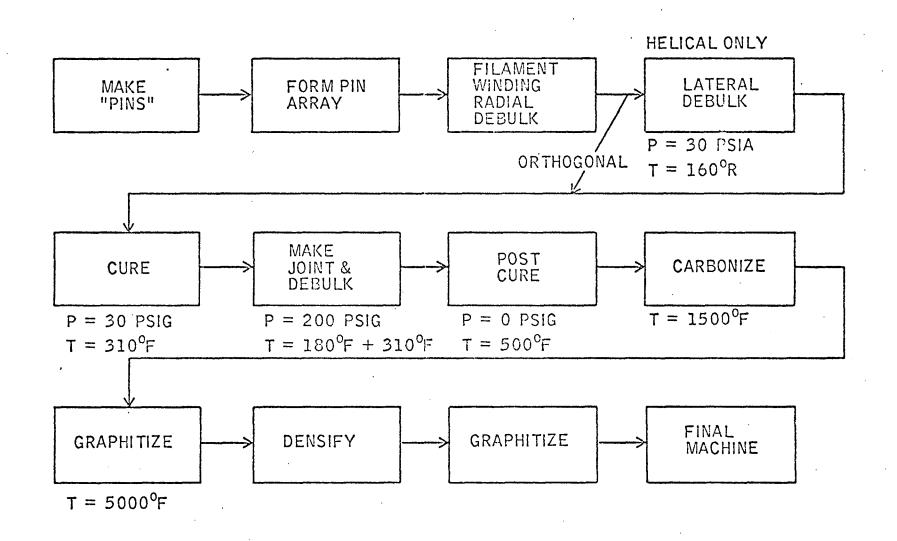
The radial axis reinforcing members are made first by molding resinimpregnated graphite yarns into long, stiff strands with diamond or square shaped cross-sections, and then cutting these strands into short radial cells, or "pins". Next, a pin array is formed by inserting these cells radially into a hollow, polystyrene foam mandrel, of the general shape of the desired part, and securing the inside ends of the cells in polyurethane resin. After resin cure the polystyrene is dissolved.

After the mandrel is dissolved graphite yarns are wound into the channels between the cells. These windings are impregnated with resin and radially compressed ("debulked") periodically during winding (Figures 5 and 6). After winding, the assembly is given a final resin impregnation.

Helical material, because of its construction, can be longitudinally compressed as well as radially. This step is called "lateral debulk" and is accomplished by expanding the part into a female mold. Figure 6 illustrates this process, which causes the part to shrink longitudinally and expand axially. When the helical part has been laterally debulked it is cured, "in situ".

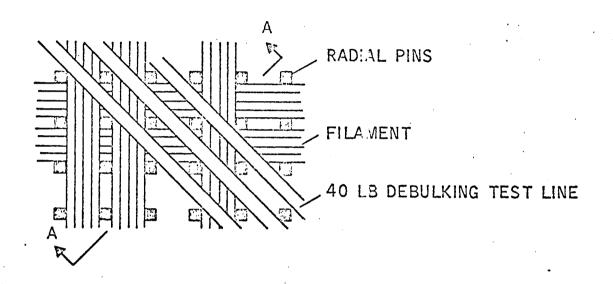
Orthogonal material is cured after final impregnation.

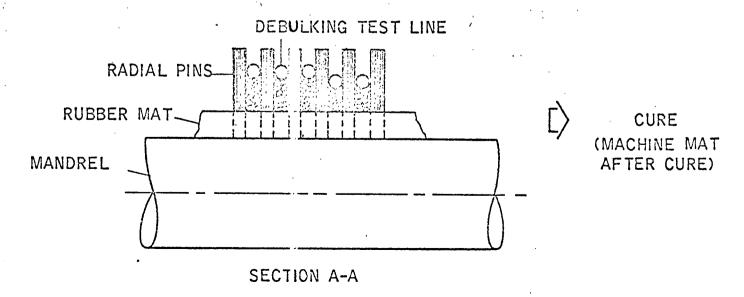
INTREMOLD III PROCESS FLOW CHART



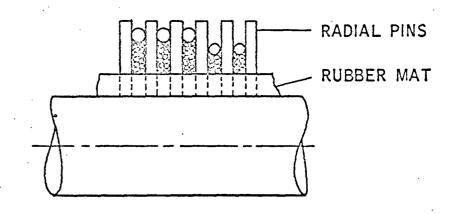
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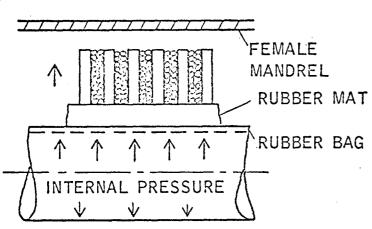
DEBULKING PROCESS ORTHOGONAL ORIENTATION

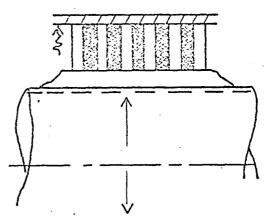


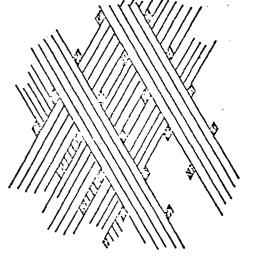


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POST DEBULK
ORIENTATION
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FIGURE 6

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After cure, the part is machined in preparation for joining to AGCarb. The AGCarb "pre-preg" (graphite fabric pre-impregnated with resin) is laid-up on the Intremold III joint surface. Depending on the type of lay-up, in-process debulking of the AGCarb may be required. This is accomplished by applying heat and pressure to the lay-up. As a final step the resin in the lay-up is cured.

The next three steps are used to process the resin from a cured phenolic to a graphite state.

After initial graphitization the specific gravity of the part is low, perhaps 1.1 or 1.2. The densification step is a series of re-impregnations, cures and carbonization which build up the density to the desired level. The impregnant used is a high carbon yield pitch. After the density goal is reached the pitch is graphitized and the part is machined to its final configuration.

Step-by-step fabrication details along with evaluation of the process are given in the subsequent sections.

PART A

MANUFACTURE THROUGH FIRST CURE OF SIX

EXPERIMENTAL INTREMOLD III CYLINDER ASSEMBLIES

ENGINEERING OPERATIONS REPORT

The Manufacture Through First Cure

of
Six Experimental Intremold III Cylinder Assemblies

Project 143,f,3 and i,2

21 January 1972

E, F, Thacher

Nozzle Extension Section

Approved:

L. A. Shurley, Manager Nozzle, Pressure Vessel and Skirt Department

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	Ortho 3 Cure Cycle

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I. INTRODUCTION

The nozzle assembly of the NERVA engine consists of a stainless steel, convectively-cooled nozzle extending from the base of the reactor to area ratio 24:1 and includes the nozzle inlet chamber and throat. At area ratio 24:1, the nozzle is joined to a fibrous graphite skirt or Nozzle Extension (NE). The NE is 4-1/2 feet in diameter at area ratio 24:1, 12 feet long, and 10 feet in diameter at area ratio 100:1, the exit.

AGCarb, a laminated fibrous graphite composite, is the preferred candidate material for NE construction. However, it may be deficient in interply strength for application in the NE forward flange area where most of the stresses occur.

Intremold III was selected as a possible alternate candidate material in this forward flange area. It is a tri-directionally reinforced fibrous graphite composite material, and potentially structurally superior to the laminated composite in interply strength. This created a new problem of joining an Intremold III forward flange to an AGCarb liner (NE shell).

Fabrication of six fibrous graphite cylinder assemblies was undertaken to provide Intremold III material and Intremold-to-AGCarb joint specimens for evaluation. These assemblies are composed of a central cylinder made of Intremold III, joined in several different ways at each end to two short cylinders made of AGCarb. The purpose of this report is to document all aspects of the design and manufacture, through first cure, of these six cylinder assemblies.

II. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

A. SUMMARY

- l. Six Intremold III cylinder assemblies were built. They were 9 in. O.D. x 12. long with a 1/2 in. thick wall. Three were helically wound (two intersecting helical winding paths; diamond shaped radial cells) and three were orthogonally wound (two mutually perpendicular intersecting winding paths; square radial cells). Each cylinder incorporated an AGCarb-to-Intremold III integrated transition joint at each end, with two joints repeated once. Most of the joints were basically variants of a lap joint. One was a butt joint. The joints are listed in Table 12.
- 2. The process through first cure used to make the cylinders consisted of six main steps:
 - a. Manufacture radial cells.
 - b. Form a cylindrical cell array.
 - c. Filament wind the array.
 - d. Resin impregnate.
 - e. Radial and lateral debulk. (lateral debulk helical only)
 - f. Cure.
 - g. Joint manufacture (some joints were made before cure).

Difficulties were encountered with the fabricability of three of the joints, with the compatibility of "B" staged Intremold III and the normal AGCarb debulk and cure pressure, and in other areas. It required essentially four cylinders to arrive at a satisfactory process and product.

B. CONCLUSIONS

- 1. Based on joint fabrication experience:
 - a. An engine-sized integrated joint between AGCarb and Intremold III can be fabricated.
 - b. The best time to make integrated transition joints is after Intremold III cure.
 - c. Eight of the ten joints attempted are fabricable on orthogonal parts, and seven of the ten on helical parts. The non-fabricable joints are Interleave (Figure 31) and Ledge Lap (Figure 32). Comb Tooth is limited to orthogonal parts.
 - d. The Interlock joint (Figure 34) is the preferred joint from a fabrication standpoint.
- 2. Without test data, no choice can be made between Helical and Orthogonal Intremold III. However, the fundamental advantages of each material, so far observed, can be stated.

a. Helical

(1) Structurally the better because of the fiber straightening and compacting imparted by lateral debulk.

b. Orthogonal

- (1) Cheaper and easier to fabricate because filament winding is more rapid and lateral debulk is not employed.
- 3. Provided that suitable full scale tooling is furnished for the process steps listed in paragraph 4, engine sized nozzle extension flanges can be fabricated of Intremold III. The process control items listed in paragraph 5 are not limits on engine-sized fabricability.

- 4. Major new tooling for engine-sized parts is required for:
 - a. Cell insertion.
 - b. Dissolving foam mandrel.
 - c. Filament winding.
 - d. Helical cylinder lateral debulk, and
 - e. Cure.
- 5. Before parts with integrated joints can be made on a production, instead of a developmental basis, the following must be accomplished:
 - a. Establish process control criteria for each process step, including criteria for;
 - (1) Resin flow
 - (2) Resin volatile content
 - (3) Resin content
 - (4) Joint filament winding.
 - Establish acceptance criteria for completed cell stock
 based on;
 - (1) Minimum acceptable density
 - (2) Void content.
 - c. Establish acceptance criteria for the cured Intremold cyclinder.
 - d. Establish acceptance criteria for the cured integrated joint.

6. Allowances of at least 0.3 in. on the I.D. and the O.D. of engine sized parts, and 0.3 in. axially, should be left for machining and machined edge heights should be >0.1 in. for Intremold III.

C. RECOMMENDATIONS

- 1. If test results indicate that Intremold III requires further development to meet NE requirements, work should be carried out in the areas of:
 - a. Improving part separation from the mold ("mold release").
 - b. Investigating inside-out molding for Orthogonal parts.
 - c. Investigating filament winding of Intremold III with pre-pregged ýarn.
 - d. Producing void-free cell stock.
 - e. Determining if cell and part resin contents and resin flow and volatile content for each process step are optimum.

III. DISCUSSION

A. THE INTREMOLD III MANUFACTURING PROCESS

1. Material Description

Intremold III is a reinforced graphite composite material. The reinforcement consists of an array of filled cells, or "pins", running transversely through the thickness and of filament windings (forming the "cell walls") laid in the channels between the pins. The composite structure is bound together, and all voids are filled, with resin and pitch, which are processed to the graphite state.

When made in cylindrical form, Intremold II is called "helical" or "orthogonal" depending upon how the pin cross-section is shaped and how the filament windings are run. Helical material has a diamond-shaped pin cross-section, and the filament windings are laid in two helixes, half slanting to the "right" and half to the "left" when the pin is viewed from the top. (see Figure 1).

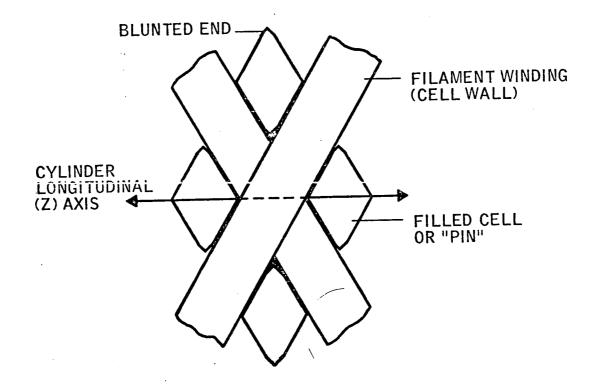


Figure 1
Helical Intremold III

Orthogonal material has a square-shaped pin cross-section. The circumferential windings are wound in a continuous helix of very small pitch, so that they are nearly at right angles to the axial windings (see Figure 2).

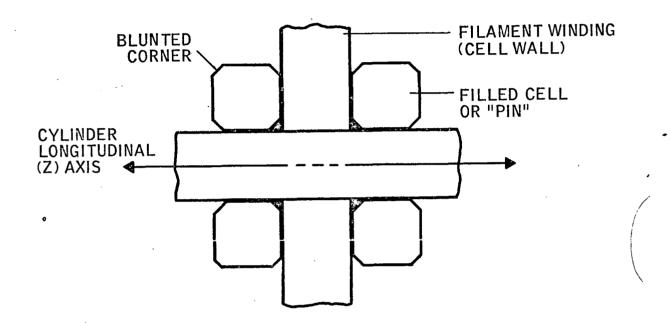


Figure 2
Orthogonal Intremold III

Blunting the ends of the diamond-shaped cells increases the amount of cell wall debulk obtainable. When the filaments are wound, the blunts form a wider channel than would exist with unblunted cells. However, they have no influence on the minimum cell wall width obtainable during lateral debulk (fiber compaction). Thus, they increase the compacted fiber density. On square cells (and on diamond cells) they increase the fiber volume at the crossover points, thus adding to strength.

2. Process Outline

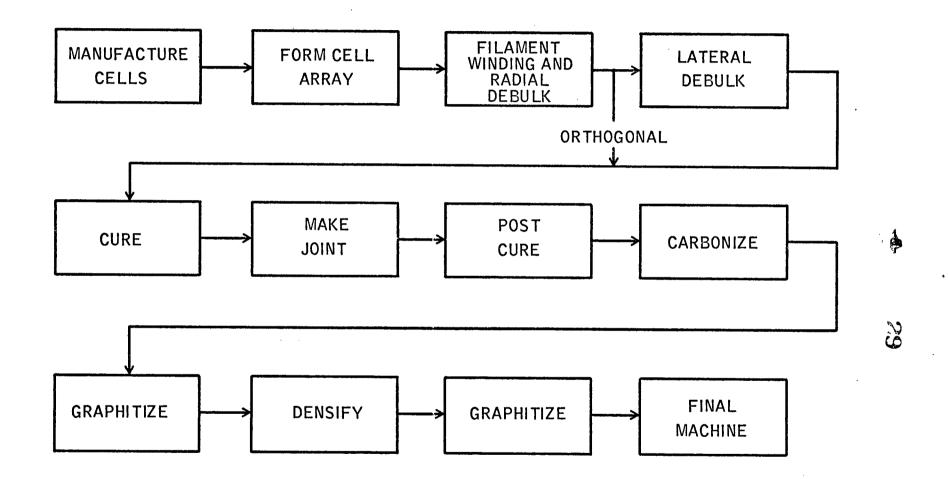
Figure 3 is a simplified flow diagram of the Intremold III manufacturing process. In the succeeding sub-sections each block in Figure 3 will be discussed in detail.

3. Manufacture Cells

The cell making process consists of three steps. First, graphite fiber strands are made by binding a bundle of graphite yarns together with resin. This is called "collimation". Next, the strands are molded into pin stock with a square or diamond-shaped cross-section. The molding process applies heat and pressure to form the cell stock and cause the thermosetting resinto harden ('cure"). Finally, the cell stock is pulled through scraping dies to deflash it and to give it its final transverse dimensions. Then it is cut to the desired length and sampled for proper density.

a. Collimation

Figure 4 shows the equipment used in collimation. The yarn creel, on the right, supplies yarn to the three teflon collimating rings, one for each collimated strand. There are four or six yarns in each strand, depending upon whether the strands are for diamond cell or square cell stock. After passing through the collimation rings, the yarn bundles move through the cylindrical heat cleaning oven where surface impurities are removed by heating in an inert gas atmosphere. After cleaning, the yarn bundles are impregnated with resin in the vacuum impregnation bath and drawn into an oven called the "staging box". In this box the resin in the yarn bundles is advanced. This dries the resin to a tacky state but leaves the strands pliable. The finished strands leave the staging box and are wound on six rectangular yarn loading frames, called "harps", which are mounted on a hexagonal take-up reel. The reel and the harps are shown in Figure 4A. The reel is driven at 1/5 to 1/6 RPM by the electric motor at its base. Each harp holds 45 26-inch strands (25 inches useable material). After the reel has been loaded, locking bars are bolted over the strands at each end of the harps and the strands connecting the harps are cut. The harps are then taken to a molding press for the second stage of the cell stock manufacturing process,



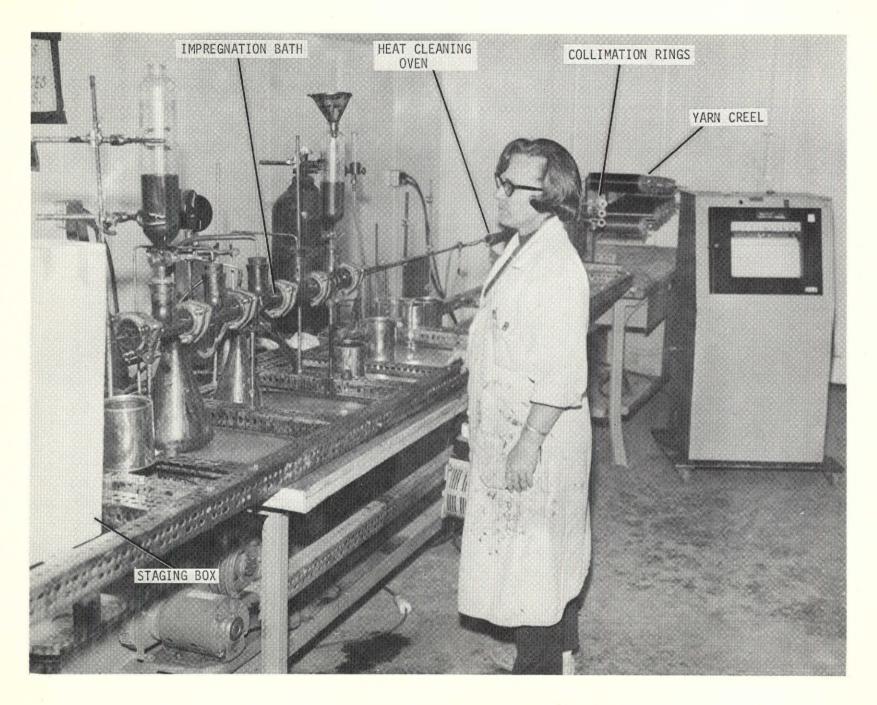


Figure 4. Collimation Facility

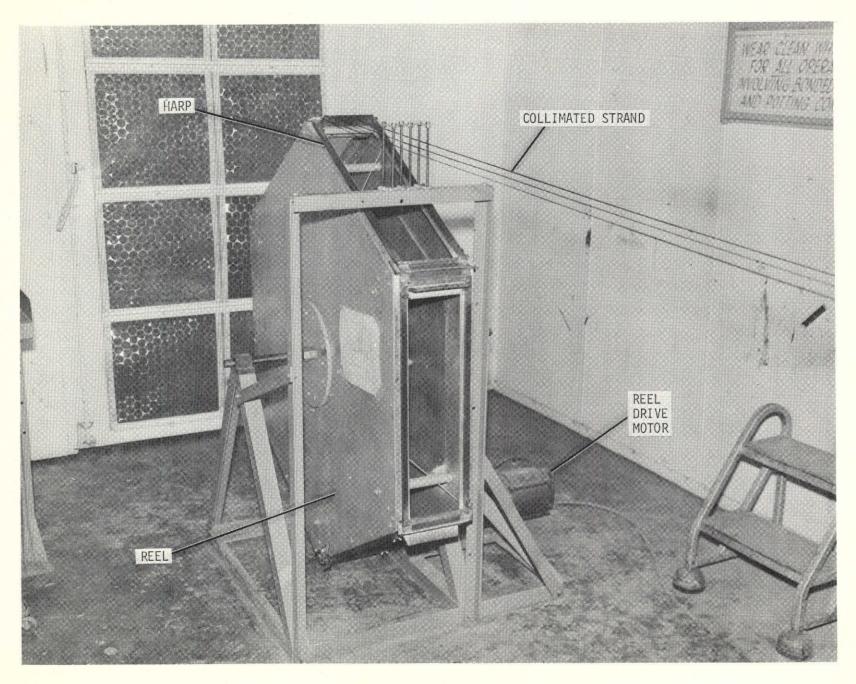


Figure 4A. Take-Up Reel

(1) Post Collimation Inspection

When the collimation run is completed (all harps have been loaded) the percent volatile content of the strands is determined from samples of all three strands. Resin content is determined by acetone extraction of the resin and volatiles. The percent change in weight is taken as the measure of the quantity involved in each case,

(2) Problems Encountered

Based on the data generated by the inspections for resin content discussed above and the assumption that this data population has a normal distribution, the strand rejection rate from this population was estimated at 72%. This is an unacceptable rejection rate. The rejection criteria used was the resin content tolerance of \pm 2% on pre-preg fabric given in specification AGC 902292. However, this criteria may be too stringent for this process. A tolerance equal to the coefficient of variation of the resin content data (31%) would yield about a 32% rejection rate. (Supporting calculations in Appendix A). The criteria should be examined in the light of the allowable resin content variation of the finished cell stock to show whether such close control of strand resin content is required.

Resin content is affected by all the parameters listed in Table 1 except the staging box and cleaning oven temperature (unless, of course, the latter is not hot enough to clean the yarn). The specific gravity of the resin affects the weight of resin per unit volume impregnated. Viscosity affects the ability of the resin to penetrate the yarn bundles. Bath vacuum affects the driving head forcing the resin into the material and helps to remove any air trapped in the yarn. Reel speed affects the dwell time of the resin in the impregnation bath, and therefore the weight pickup of resin per unit length. An additional factor not listed in Table 1 is the tendency of the filler in the USP-39 impregnation resin to settle over the period of a collimation run. This reduces the resin-to-filler ratio in the impregnated strands. The magnitude of this effect is not known and should be evaluated. In addition to resin content the optimum resin flow and volatile content for strand impregnation should be established.

TABLE 1
COLLIMATION PROCESS PARAMETERS

Process	Collimation
Step	Parameters .
Yarn Speed	34 in./min (1/5 reel rpm) - Helical strand stock 28 in./min (1/6 reel rpm) - Orthogonal strand stock
Heat Cleaning	T = 1000° <u>+</u> 150°F N ₂ flow = 15 <u>+</u> 5 CFH
Impregnation	Resin sp, gr = $1.150 \pm .005$ and viscosity = $975 \pm .75$ CPS bath 1 and 5 ambient pressure, 2 and 4 at 15 in. Hg vacuum, and #3 at 20-25 in. Hg. vacuum. (Viscosity measured by RVF Brookfield meter, Spindle #1, 10 r.p.m at resin temperature $72^{\circ}F$)
Staging Box	T = 250 <u>+</u> 10°F
Helical Rate of	of production = 2/3 harp/hour te = 4/5 harp/hour

24

(3) Scale-Up

No changes in the collimation process are required to produce engine-sized Intremold III parts.

b. Molding

Before the actual molding, the loaded harps are heated to $180^{\circ} \pm 5^{\circ}$ F in an oven for an hour. This treatment advances the resin toward cure ("Staging") and therefore reduces resin flow during molding (resin volatiles content is also reduced). This improves mold release, (Reference (8)).

After staging, the harps are taken to the molding facility conveyor table, shown in Figure 6, to be assembled into molds. The female mold dies are sized and shaped to produce the proper square or diamond geometry, as Figure 7 illustrates.

The lower dies and spacers are assembled first. A loaded harp is placed over this sub-assembly and the strands are fitted into the die cavities. Finally the upper dies and spacers are fitted over the strands (Figure 5) and the entire assembly is bolted securely together. Three assembled molds are placed in the press at a time. Figure 8 shows molds in the press.

Molding pressure and platen temperature are controlled from and displayed on the control panel shown in Figures 6 and 8. A hydraulic ram applies force to the upper platen to compress the three molds in the press. The amount of compression, called "mold deflection", is taken to be the interplaten distance. Mold deflection is continuously measured by a dial indicator.

The molds are heated by conduction from the platens which themselves are electrically heated.

The entire molding cycle, from the beginning of heat-up to the end of cooldown, requires about two hours. When the cycle is completed the harps are removed from the molds as shown in Figure 8.

Molding cycle parameters are listed in Table 2.

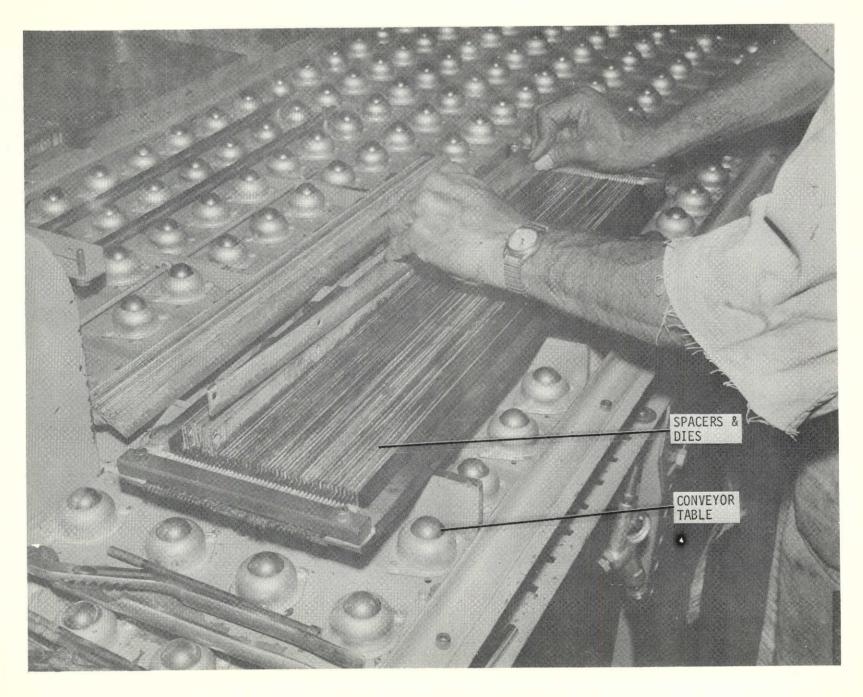


Figure 5. Assembling A Mold

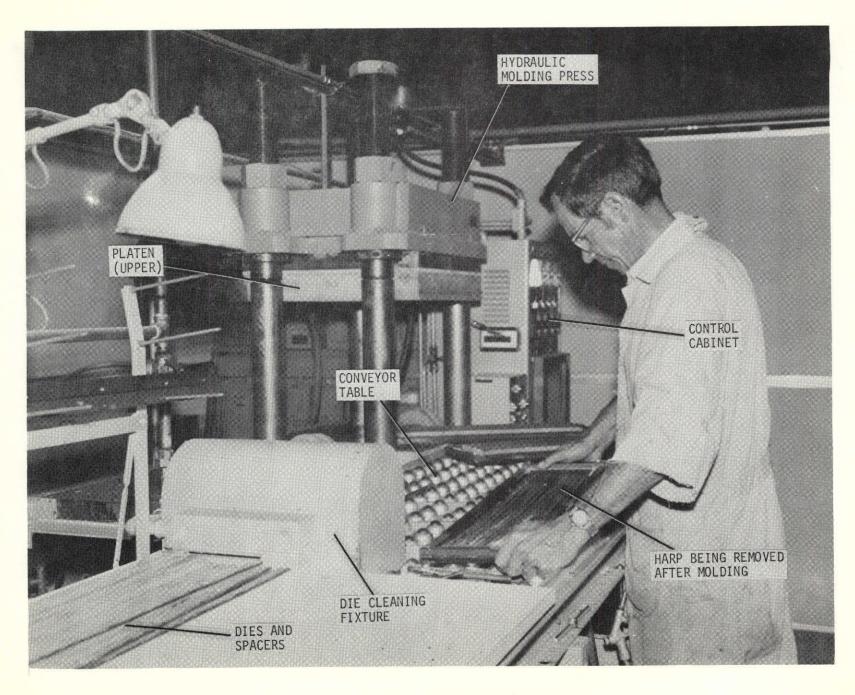


Figure 6. Molding Facility

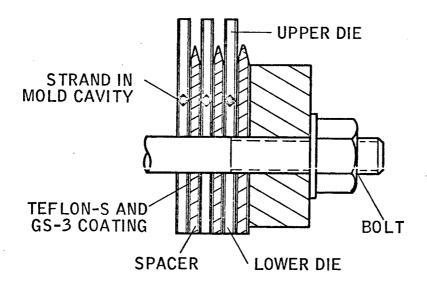


Figure J
Mold Assembly Diagram

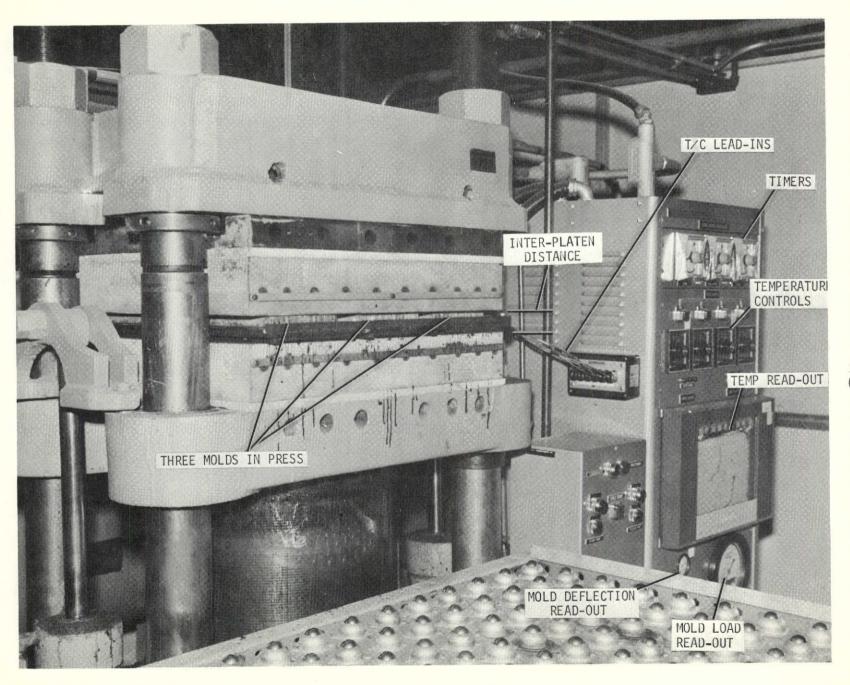


Figure 8. Molds In Press

TABLE 2

MOLDING PROCESS PARAMETERS

SQUARE AND DIAMOND CELLS

p 						
Process	Cell Stock Molding					
	Parameters					
Step						
Oven Stage 60 min at 180° ± 5°F						
Molding	Time(N	1) Temp(°I	F) Min Load(T)			
Pre-heat	15	200	As required to maintain deflection.			
Cure 1	15	240				
Cure 2	60	310	der rection.			
Cool	8	<140				
Final Deflection	1,524 Diamond Cells					
(in.)	1,514 Square Cells					
Process Rate 1 1/2 molds/hour = 67 strands/hour = 1608 in./hr						

(1) Post Mold Inspection

No measurements are made on the cell stock immediately after molding. A visual inspection is performed to detect stock damaged by improper mold release. Void free cell stock can and should be obtained. Rejection criteria related to void content should be established. See paragraph c.(2), below for discussion.

(2) Problems

No data on the correlations between resin content and cell mechanical properties, or between resin content, resin flow and volatile content, and the molding process variables, are known to exist. Such correlations should be established to provide assurance that present control parameters are proper and before a specification for the molding process is written.

The resin content of the cell stock (and of the composite cylinder) is important. Too little resin lowers cell strength because the fibers are weakly bonded together; the composite strength therefore approaches that of a bundle of unsupported fibers. Too much resin means that there is insufficient reinforcement; the composite strength approaches that of the matrix alone. In between these extremes the strength rises to a maximum near 30% resin (by weight) (Reference (10)). Figure 9 illustrates these effects near this optimum resin content. Excess resin also causes matrix cracking and separation between the yarn and the matrix because of shrinkage, which increases with resin content (Reference (14)). The resin content which produces the least shrinkage may not correspond to that which is optimum with respect to strength (Reference (10)).

During the molding process the heat and pressure applied to the strands in the molds first cause the resin to flow, and then to cure. Excess resin is squeezed out of the mold and hardens into "flashing" which must be removed from the cell stock during the deflashing and sizing process which

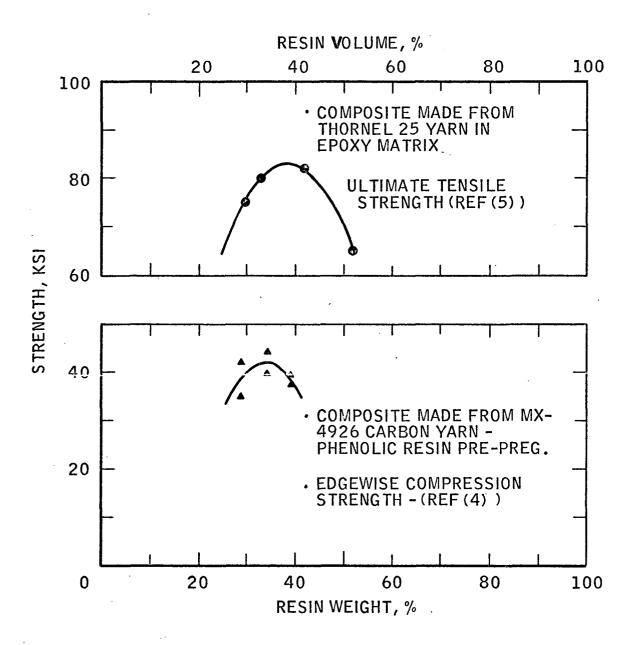


Figure 9
Effect of Resin Content on Strength

follows molding. Resin squeeze-out lowers the cell stock resin content from its post-collimation level of $54^{1}\%$ to about $35^{2}\%$. This may be compared with the resin content of AGCarb after first cure of about 32%.

Inspection of Figure 9 indicates that cell resin content may be near optimum. However, as the first paragraph points out, this needs experimental verification. This verification should be made from the standpoint of optimum performance of the graphitized cell stock.

In the past, difficulty has been encountered in stabilizing the adhesion of the baked-on Teflon S die mold release coating. Reference (8) details efforts to eliminate this problem. This difficulty was not encountered while making the present cylinders. However, the supplementary sprayon teflon coating, GS-3, had a usage rate much higher than anticipated. This usage rate increased the manufacturing costs of the cell stock.

The tie-bolts holding the molds together have a high failure rate caused by stripping of the bolt heads. This problem could be minimized by establishing proper assembly bolt torque values. Mold bolt-up would then be accomplished with a torque wrench.

Cell stock resin voids occur during molding.

Figures 10 and 11 are photomicrographs of cured and sized cell stock. The black shapes in the cell in Figure 10 are voids in the resin. The cell shown in Figure 11 has very few voids. The most desirable condition is to have no voids. Voids lower cell effective strength by acting as stress risers and crack starters

¹ Mean of data from resin acetone extraction performed after collimation.

² Mean of samples from cell lots 2003-100, 101 and 102 taken after sizing and cutting.

³ Estimated from pre-preg data.



Figure 10. Diamond Cell - Lot 2003-102 100X As Cured

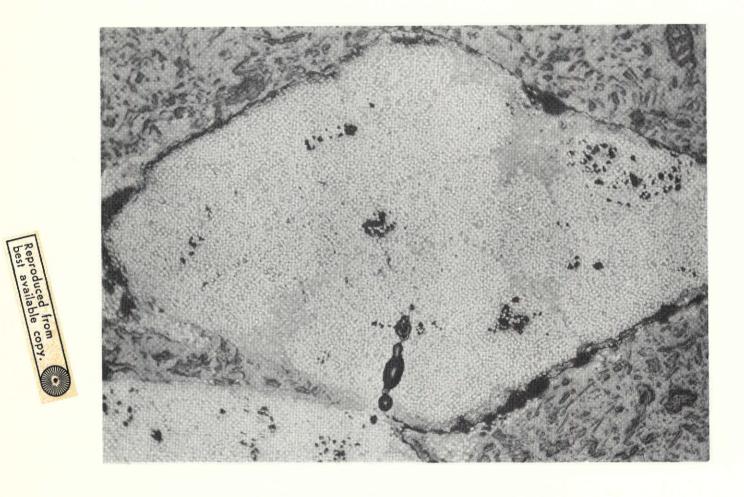


Figure 11. Diamond Radial Cell - Lot 2003-102 100X As Cured

(Reference (3)). Of the cell stock sampled and photographed, the orthogonal samples seemed to have the fewest voids.

Oven staging prior to molding should be controlled to a specified resin flow and a specified resin volatile content. These must be determined.

c. Sizing and Cutting

To deflash the cell stock and to bring it to its final, blunted, cross-sectional dimensions, each loaded harp is placed on brackets on the carriage of the sizing machine, Figure 12. This machine is a modified surface grinder mounting two sets of upper and lower tungsten carbide dies of the proper blunted diamond or square shape. The molded cell stock is sized (and deflashed) by pulling it through the two sets of dies. This is done by manually operating a wheel (not shown in Figure 12) which is geared to the carriage. Cell transverse dimensions are taken by micrometer after each carriage traverse.

Cutting the cells to length is done in the cut-off machine shown in Figure 13. After setting the machine's index bar for the proper cell length, the cell stock is removed from the harp and fed through the rollers which advance it automatically to be cut. The abrasive cut-off wheel moves across the cell stock, severing cells of the desired length. The range of length settings, without modification to the machine, is .75 to about 1.25 in.

After cutting cells are stored by lot in marked containers.

Sizing and cutting rates are shown in Table 3.

(1) Inspections

In order to characterize the results of the cell making process, cell stock density was determined by subjecting samples from each lot to a water-immersion test. No cells were rejected on the basis of low density because no density rejection criteria existed. Under regular production conditions

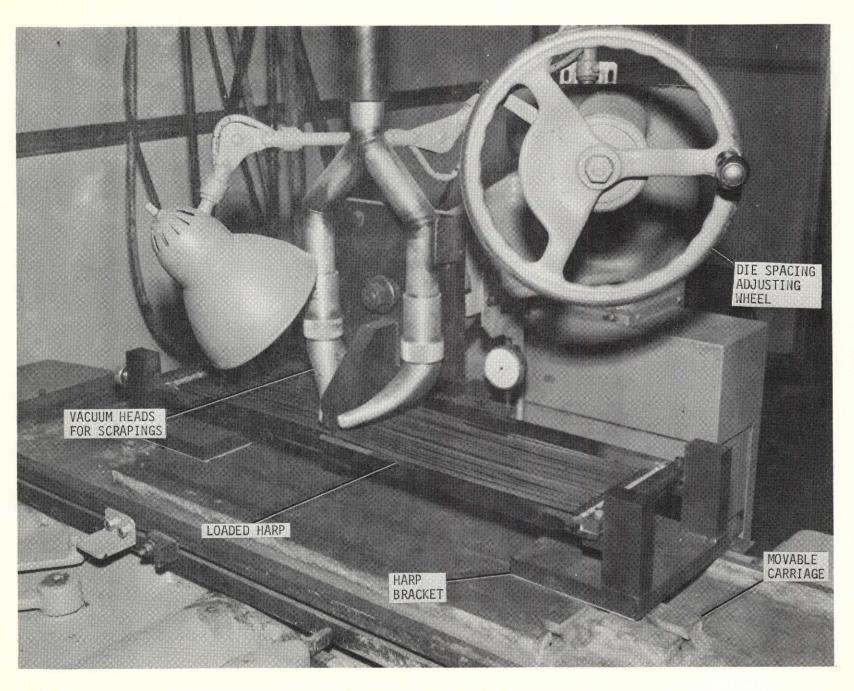


Figure 12. Sizing Machine

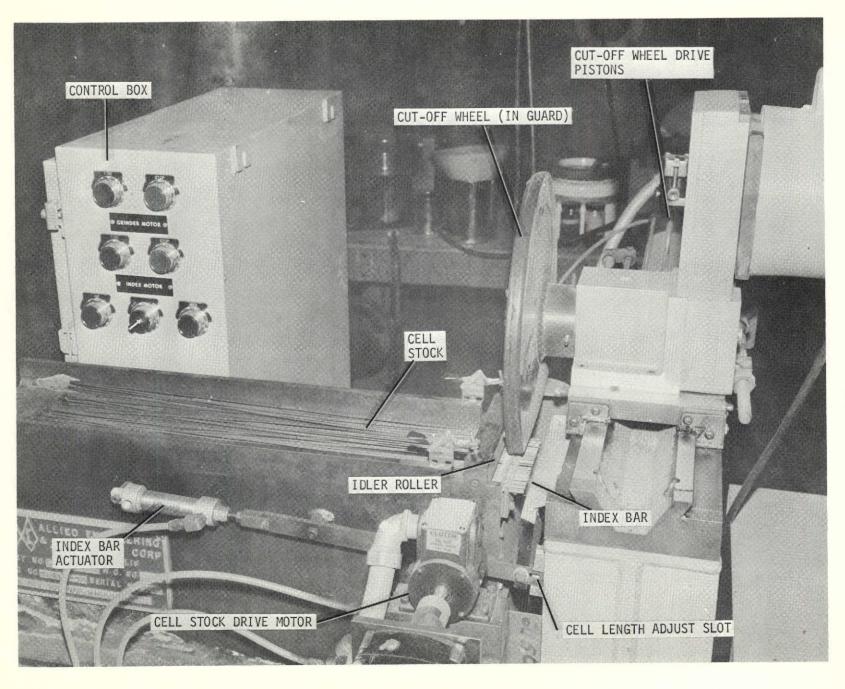


Figure 13. Cut-Off Machine

TABLE 3
SIZING AND CUTTING PROCESS RATES

Process	Rate		
Sizing	4140 Strand-in. hr		
Cutting	5400 <u>Cells</u> hr		
Density Check	18,000 ^l <u>Cells</u> hr		

 $^{^{}m 1}$ Based on .75 inch long cells.

the density of each cell would be checked after cutting by immersion in a solvent solution of pre-selected specific gravity. Floating cells would be rejected. Thus a density "floor" would be established for cell stock production.

Although not done during cylinder production, sampling for proper cell length should be done after the cutting operation.

(2) Problems

The tungsten carbide dies wear rapidly, necessitating their being sharpened about every twelve harps.

A density reject criterion must be established. This would be done as part of a program (recommended in the previous section) to determine the optimum cell resin content as a function of the various process variables.

Optimum cell density would be the density resulting from the optimum resin content. This resin content may be near 30% by weight, as has been suggested. If it is assumed to be 32% (used earlier as the estimate of the resin content of cured AGCarb), and 1.30 gm/cc is taken as the density of cured USP-39 (the resin used to make the radial cells, see Reference (16)), and 1.28 gm/cc as the density of the graphite yarns in the cell, the theoretical (voidless) cell density is 1.29 gm/cc (see Appendix B for this calculation). The mean cell density actually measured (by water immersion) was

Square cells 1.325 gm/cc

Diamond Cells 1.292 gm/cc

Compared to the calculated value these figures indicate that the values of the cell processing variables used for the cylinders may be near optimum, except for the elimination of voids.

(3) Scale-Up

The engine-size flanges will require cells about two inches long. The cut-off machine index bar must be modified to produce cells of this length.

4. Forming Cell Array

a. Foam Mandrel

The first step in forming the cell array is to make a hollow cylinder of proper wall thickness, diameter and length. The cylinder is machined from a polystyrene floatation billet, and is therefore called a "foam mandrel". The foam mandrel is mounted between the headstock and tailstock of a cell insertion machine (a modified lathe). This is shown in Figure 14. The foam mandrel shown is the size used for the "orthogonal" cylinders. Its wall thickness, 0.D. and punched length are about the same as the finished cylinder. The foam mandrel punched length for the "helical" cylinder is longer and has a smaller 0.D. than the finished cylinder, although its wall thickness is about the same. The helical mandrel must be sized in this fashion to allow for the length decrease and diameter increase of the cylinder during lateral debulk (see Section III.A.6).

b. Cell Insertion

Next, the cells are loaded into the cell insertion head feeder bowl ("Syntron Bowl") (not shown in Figure 14). The cell insertion head is mounted on the lathe cross-head. Cells travel from the feeder bowl into the escapement chute which supplies the cell magazine by gravity feed. The magazine is automatically operated to line up the cell with the insertion plunger before the inserting stroke. The insertion plunger and the hole punch are actuated together. They are positioned by the coupled rotating and translating action of the indexing gear on the head stock and the lead screw, which drives the crosshead. The indexing gear is operated by a ratchet and held in each new position by a locking pawl. The entire mechanism is driven by compressed air.

The maximum insertion rate is about 60 cells/min. Each helical cylinder contains about 43,000 cells and therefore requires about 12 hours to insert the pins for one cylinder. Each orthogonal cylinder contains about half that many cells and therefore requires about 6 hours for insertion. In actual practice, the automatic insertion feature was not used consistently. The reasons for this are discussed in subsection b., below.

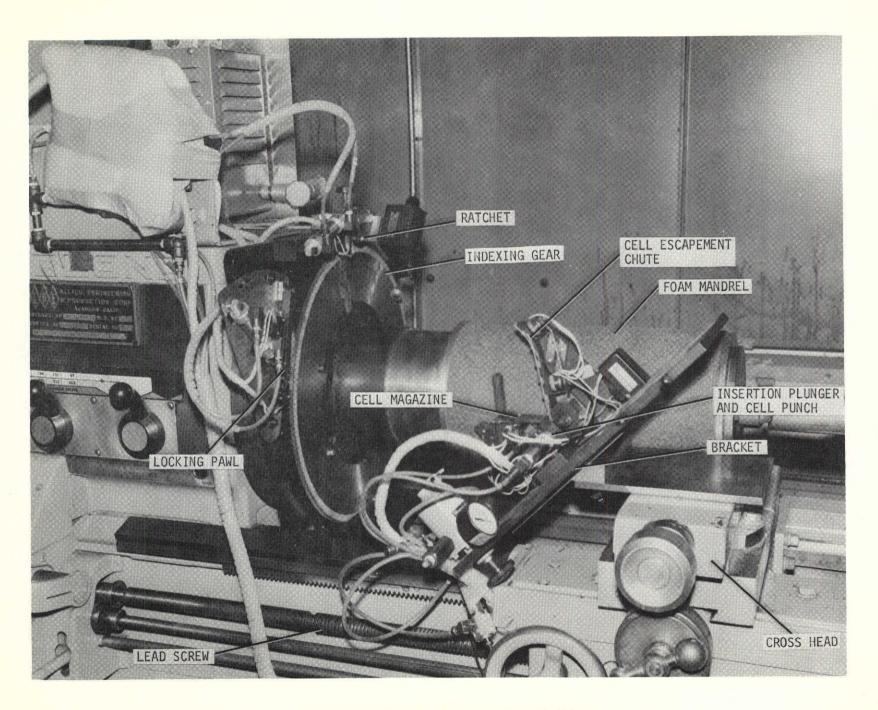


Figure 14. Cell Insertion Machine

TABLE 4

CELL ARRAY PROCESS PARAMETERS

Process	Helical	Orthogonal				
Carriage Feed	14 threads in.	8 threads in.				
Lay-up Head Dist. from Mandrel	.05 in,	.05 in.				
Resin Backing	<u>-</u> .	1050 gm, Flexane 95 & 3 layers style 181 GRP				
Flexane Cure	12 hours at RT	12 hours at RT				
Spin Casting	25 rpm for 240	25 rpm for 240 min.				
Glass Reinforced Plastic	2 hours at 140°	2 hours at 140°F				
Max. Ins. Rate	60 cells/min.	60 cells/min.				
Array Prod. Rate	About 1000 Resi	About 1000 Resin backed cells hour				
Foam Mandrel		Dow Chemical "Styrofoam FR" ¹ (Polystyrene foam)				

¹ Not original foam, but improved version.

c. Resin Backing

When cell insertion is complete the resin backing is cast. The foam mandrel wall thickness is less than the cell length, so that about 0.125 -.150 in. of the cell protrudes through the inside surface of the mandrel. Then, while the mandrel is still in the insertion machine, the outside of the mandrel is taped to secure the cells during casting and polyurethane resin is spin-cast on the inside of the mandrel to hold the cells inner ends. After this rubber-like resin has cured, glass reinforced plastic is laid up on the polyurethane's ID to give it rigidity.

The cell array in this condition is shown in Figure 15.

d. Dissolve Foam Mandrel

As a final step the polystyrene foam is dissolved by washing it in methylethyl ketone (this usually required about three washings in MEK).

Cell array forming process parameters are shown in

e. Inspections

Table 4.

The foam mandrel is inspected after machining for proper dimensions and damage. After cell insertion and before casting the resin backing, the mandrel-cell assembly is inspected for foam damage, and to insure all cells are flush with the mandrel outer surface. After the resin backing has cured and the foam has been dissolved the finished cell array is inspected for missing cells (these can be manually inserted during the filament winding process).

f. Problems

Malfunctions of the indexing features of the lay-up machine and trouble with the cell feed caused most of the cell insertion for the helical cylinders to be done by hand. The lay-up machine was used for punching the foam mandrels and for spin-casting the resin backing. Lay-up of the orthogonal

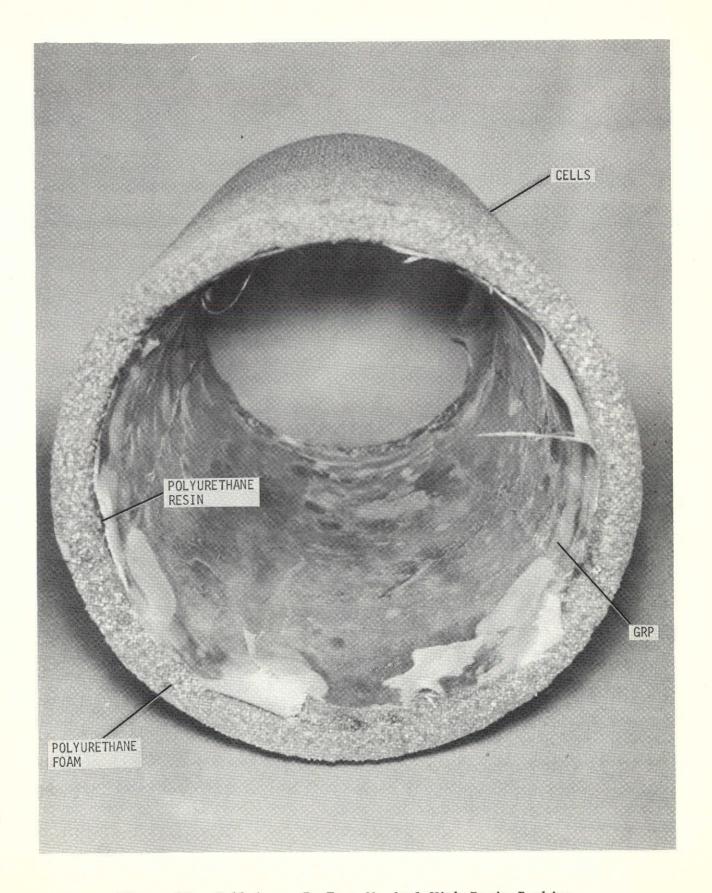


Figure 15. Cell Array In Foam Mandrel With Resin Backing



cylinders was done completely by hand. This came about because of the design of the only available square-cell lay-up head. The punch on this head, and its guide tunnel, were tilted at a 45° angle to the longitudinal axis of the cylinder. To compensate, the entire lay-up head was mounted on a bracket which tilted it up at an angle of 45° to the same axis (See Figure 14). Then the sides of the punched holes lay in the circumferential and axial directions. Unfortunately, because of the fixed spacing between punch and inserter, tilting the lay-up head caused misalignment between the insertion plunger and the punched holes. Hence, manual insertion had to be used.

The coarsness of the foam billets used to make the cylinder cell arrays caused small non-uniformities in the inter-cell spacing. In addition, the coarse surface foam showed a tendency to collapse during punching. This particular foam was chosen because it can readily be chemically dissolved. A substitute polystyrene form of much finer texture has been found, and will be used in any future projects.

g, Scale-Up

Cell array forming for engine-sized nozzle extension flanges requires a completely new lay-up machine. This machine must be capable of supporting and indexing foam mandrels of about 57-inch OD. The cell insertion head should be capable of using cells up to two inches long. To allow lay-up of flange contours the length of stroke for the punch and inserter should be variable. Perhaps it could be cam operated (see also sub-section III.A.5.c).

It has not been verified that foam billets of the size required to produce engine-sized mandrels exist. If they are not available, smaller billets might be glued together to provide the requisite size.

An MEK resistant tank, big enough to hold the foam mandrel, and associated handling fixtures must be available for dissolving the foam mandrel.

E. F. Thacher 21 January 1972

5. Filament Winding

Winding the yarn reinforcement on to the cell array is accomplished with the array mounted in a filament winding machine. An operator winds the filaments (dry graphite yarn) by hand into the channels between the cells. The yarn is supplied from spools mounted in a creel located above the filament winding machine. The machine assists by rotating the array in response to a foot treadle.

After six or seven layers have been wound, resin is applied to the yarn with an injection gun fitted with a hypodermic needle. After each resin application the filaments are radially debulked (compacted). Fifty-pound test fishing line is wound tightly into the channels between the cells. This produces a higher radial fiber density, increasing strength. Figure 16 shows the fishing line being wound on the array. After each debulking this line is removed. This process is repeated until the array has been filled.

After winding, the cylinder is given a final resin impregnation under pressure in an autoclave. To dry and stabilize the impregnated resin the cylinder is staged in an oven for several hours at or below 180°F (with a correspondingly longer time at lower temperatures). Table 5 gives staging times and temperatures for each orthogonal cylinder and Table 6 gives them for each Helical cylinder. The basting procedure is explained in Section 6. All other filament winding process variables are shown in Table 5.

a. Inspections

The present process incorporates no measurements or other inspections (other than visual) during or after the filament winding process. However, for this project the weights of the array, the yarn wound on it, and the final wound weight of the resin-backed cylinder were recorded for four of the cylinders. These measurements were taken to estimate the resin pickup during the filament winding process.

b. Problems

Filament winding is the slowest single process in the fabrication of Intremold III. Helical cylinders require about 330 man-hours and

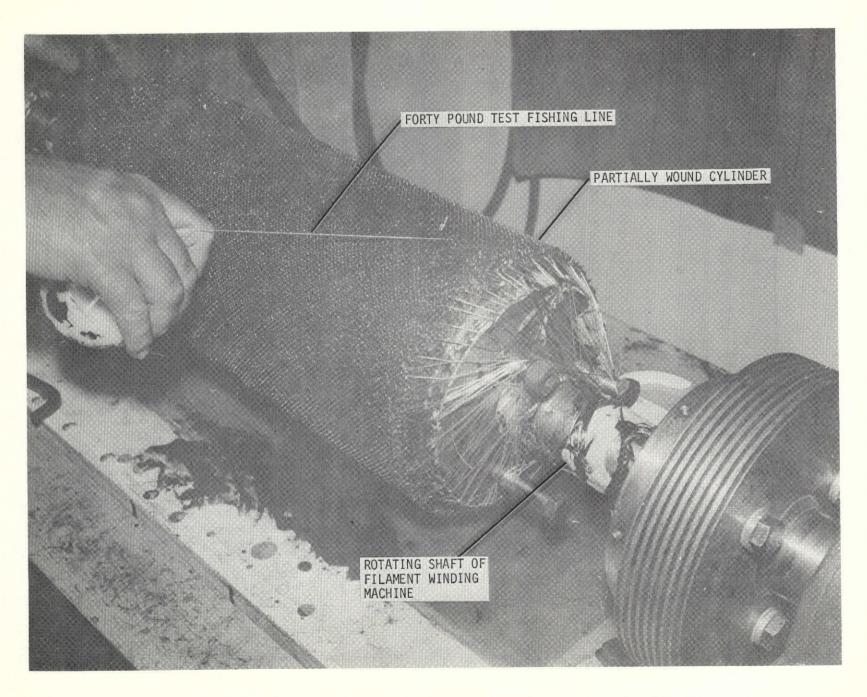


Figure 16. Radial Debulking

TABLE 5
FILAMENT WINDING PROCESS VARIABLES

Impregnate & Debulk Interval		Alternating every six or seven yarn layers		
Debulk Fishing	j Line	50 lb. Test		
Resin Parameters		200 gms USP-39 (Filled) (Resin mix F556K) diluted with 20 gms isopropyl alcohol		
Final Impregnation Parameters		95% Volume F566K and 5% volume isopropyl alcohol. Draw 27 in. Hg vac 15 min., then impregnate 200 psig 120 min.		
Number of yarns/pass		Orthogonal cylinder: 4 Helical cylinder: 3		
Oven	Time(hr)	Ortho 1 6	Ortho 2 15	Ortho 3 24 8
Stage	Temp(°F)	150 <u>+</u> 5	150 <u>+</u> 5	150 <u>+</u> 5 180 <u>+</u> 5

cylinders about half that (because the circumferential winding may be wound rapidly). Automation of the filament winding process would provide significant cost and time savings.

Weighings to determine cylinder resin pick-up and overall resin content were instituted for this project. These revealed that resin content was about 44% after filament winding (Appendix B). Since sufficient data was not available approximate calculations were made of the resin weight pick-up during autoclave impregnation. These indicated that it may be small. If this is the case this process step could be eliminated.

Whether 44% is the optimum resin content for this point in the process, or what the allowable resin content variation should be at this point, should be determined. This determination, as for cell stock, should be based on the optimum performance of fully graphitized and densified Intremold III material.

The use of pre-pregged yarn for filament winding would help to resolve the resin content problem and would eliminate in-process impregnation and possibly also the final impregnation after winding. Probably pre-pregged yarn would also be less subject to breakage during winding than the dry yarn is. It would therefore be better for use with automated winding than the dry yarn.

During hand winding it is difficult to keep the filament windings under tension, and therefore straight. This is not as important for helically wound cylinders as it is for orthogonally wound cylinders. Good filament tensioning and straightening for helically wound cylinders takes place during lateral debulk, discussed in sub-section 6. The orthogonally wound cylinders do not undergo this process. The circumferential windings in these cylinders, which can be tensioned as they are continuously and rapidly wound, also are not a problem. But the axial windings, which are laid by hand in the axial channels and make a sharp bend around each end pin (if they do not break and have to be taped down) are relaxed. Figure 17 illustrates conceptually how this problem might be solved by the use of anchor pins.



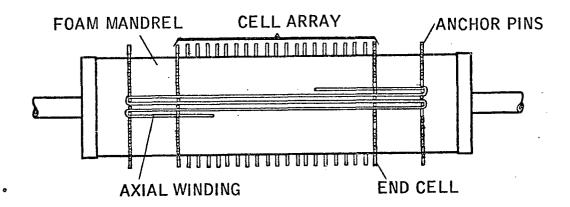


Figure 17
Concept for Orthogonal Axial Winding Tensioning

c. Scale-Up

Engined-sized flanges will require a completely new and much larger filament winding machine. It seems desirable from an economy and space-saving standpoint that the functions of filament winding and cell array lay-up be accomplished by a single, dual purpose machine. This machine would have one rotating member to turn the foam mandrel or cell array, and appropriate attachments for winding or cell insertion. It should incorporate automation to the maximum extent possible.

The advantages sought with this dual-purpose concept should be balanced against the lack of flexibility it would impose on the manufacturing schedule. It will prevent overlapping the cell insertion and filament winding processes for different parts.

6. Helical Cylinder Lateral Debulk

The construction of helical Intremold III allows an additional cell wall compaction of this material after the in-process radial debulking performed during filament winding. This process step is called "lateral debulk".

Since it would inhibit the dimensional change necessary to debulk, the resin backing must be removed by machining. Sufficient strength to withstand machining loads is obtained by staging the completed helical cylinder in an oven for several hours at or below 180°F. This staging is also required to dry the resin. The cylinder is supported in the oven so that it can be revolved about every 15 minutes. During the first three hours in the oven the cylinder is basted with resin. The purpose of revolving and basting is to reduce the loss of resin caused by flow during the initial three-hour period. Figure 18 shows cylinder Helix 1 in the oven (the reason Helix 1 appears in two parts in Figure 18 is given in sub-section III.E.1).

After staging, the cylinder is placed in a resin casting and machining fixture to support it during removal of the resin backing by machining. This fixture is shown in Figure 26. First, an ordinary single-point tool is used to remove the glass reinforced plastic. Flexane removal is accomplished by a "rubber cutting tool", shown in Figure 25. The rubber cutting tool has a knife edge, rather than a single point. It removes material like a potato peeler does - by a paring action. Thus it avoids the tendency of conventional single-point tools to grab and tear rubber-like materials.

The next step is to place the cylinder on the lateral debulk fixture. This fixture consists of a male mandrel, with an inflatable butyl rubber bag over it, an expanding female mold made of sheet metal and bonding clamps, two

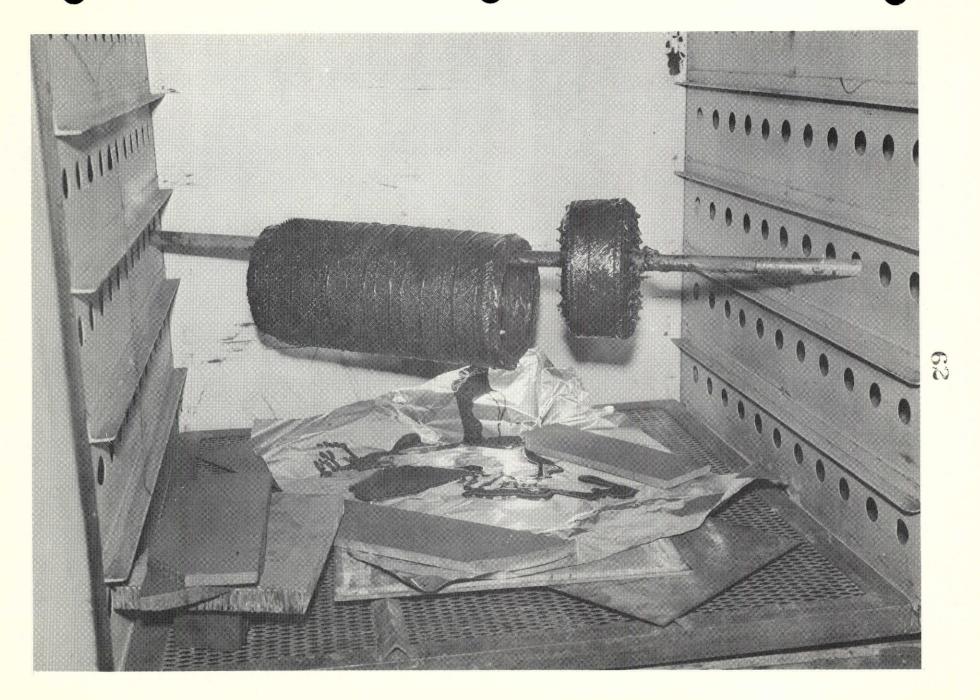


Figure 18. Helical Cylinder #1 Staging Before Lateral Debulk

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end sleeves and end ring nuts, two spacers, a rigid female mold, and an oven made of a foil-lined cardboard box with a heat gun for a heat source. All this equipment, with the exception of the oven and the expanding female mold, is shown in Figure 19.

The cylinder is fitted over the butyl rubber bag, and the expanding mandrel is placed over the cylinder. The end sleeves are installed at each end of the cylinder (see Figure 19), followed by the spacers and then the ring nuts. The entire assembly is placed in the oven and the temperature brought to about 160°F with the heat gun. Air at about 30 psig is supplied to the inside of the butyl rubber bag. The expansion of the bag forces the cylinder slowly out into the female mold. As the cylinder diameter increases, the length decreases. The ring nuts are turned to keep the end sleeves in contact with the cylinder ends. Pressure is released frequently (perhaps 3 or 4 times for a 9 in. 0D) to allow the bag to reseat under the sleeves. This process is repeated several times (perhaps 8 or 10 times for a 9 in. 0D) with the expanding mold set for larger diameters each time. When the final cylinder outside diameter (in this case about nine inches) required for this process step is nearly reached the expanding mold is replaced by the rigid female mold (of the proper inside diameter). Forcing the cylinder against this mold is the final step in the debulk process.

Lateral Debulk Process Parameters are shown in Table 6.

Figure 20 illustrates the structural features of the helical material which allow lateral debulk. Figure 20 also shows the lateral fiber compaction obtained by this process. Figure 21 shows how the axial and hoop dimensions change with final helix angle.

During filament winding, the blunting shown in Figure 20 increases the filament channel ("cell wall") width over that obtainable with unblunted cells. However, blunting does not increase the minimum channel width obtainable by lateral debulk. It therefore increases the debulk factor (a measure of the lateral debulk obtained: the ratio of the as wound cell wall width to the debulked cell wall width) over that obtainable without blunting. Blunting

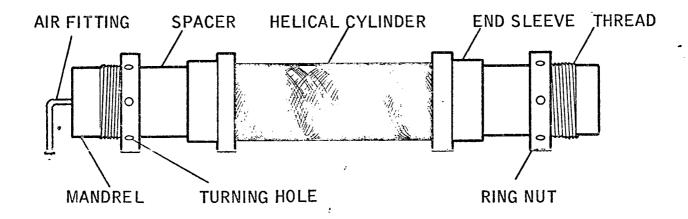
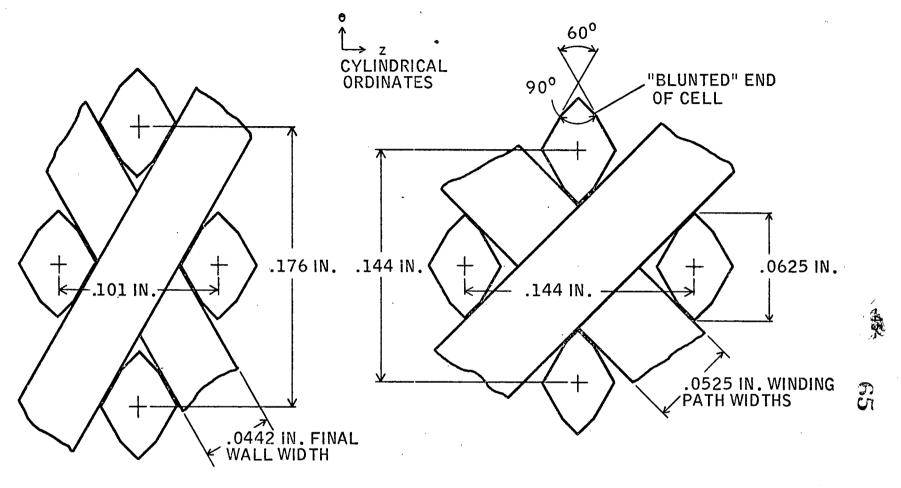


Figure 19
Lateral Debulk Fixture



AFTER DEBULKING

BEFORE DEBULKING

AREA OF RADIAL CELLS EQUALS 20%

 $r_a = \frac{\text{TOTAL CELL AREA}}{1}$ **OD CYLINDER AREA**

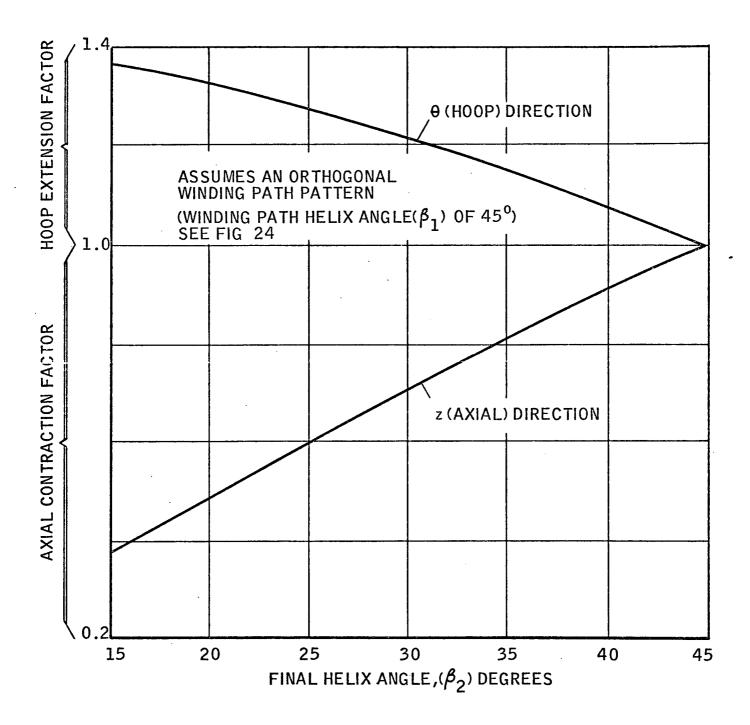


Figure 21
Overall Shape Changes During Lateral Debulk

also provides increased area for the filament wound yarns at the winding intersection points. (This is the reason that square cells are also blunted). This strengthens the composite at these intersections. Figures 22 and 23 show the theoretical effect of cell blunting and cylinder area ratio on lateral debulk. Figure 24 defines cell geometry.

Figures 20, 21, 22, 23 and 24 were taken from References (a) and (b).

a. Inspections

The helical cylinders were visually inspected, weighed and their outside diameter, length, and wall thickness determined after lateral debulk.

b. Problems

Compared to the cell lay-up machine, for instance, the lateral debulk fixture is primitive. However, only three difficulties with the process were encountered: poor mold release, less than expected lateral dimension control, and, and in one case, the bag failed to stretch sufficiently to bring all the cylinder into contact with the rigid female mold. (The resulting non-symmetrical cylinder shape was later corrected).

Poor mold release from the rigid female mold was observed in all three helical cylinders. This manifested itself in patches of the top layer of filament winding yarns being pulled up over the top of the pins and in difficulty extracting the cylinder from the mold. This problem was significantly lessened by the use of grafoil in place of polyethylene film as a mold release agent on the inside surface of the rigid female mold.

Lateral dimension control was observed to be about $\frac{+.25}{-0}$ inches, rather than the \pm .03 inches assumed during initial cylinder design. However, this problem was encountered early and was taken into account on later helical cylinder drawings. (The length of orthogonal cylinders conforms to these tolerances also.)

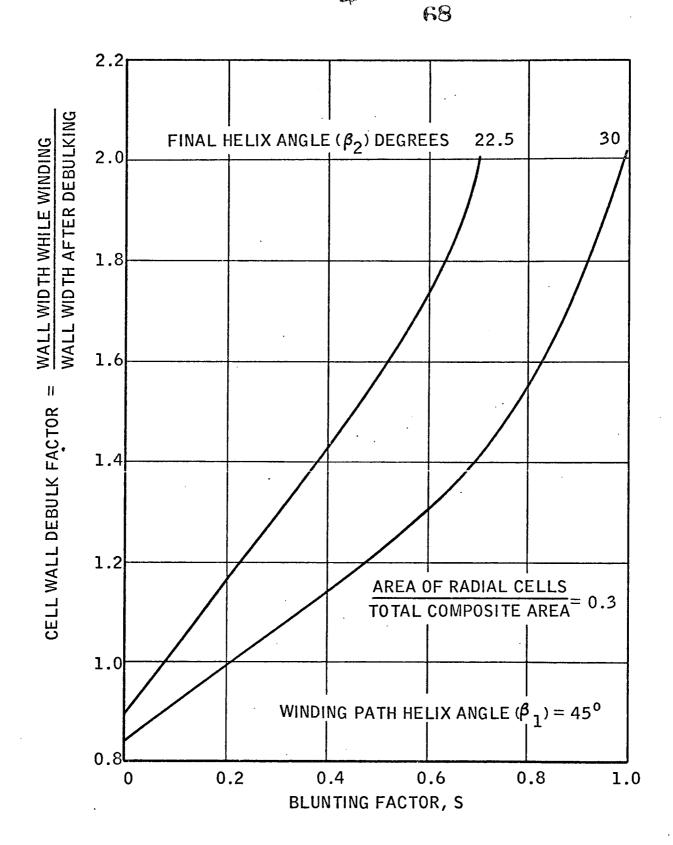


Figure 22 Effect of Cell Blunting on Debulk Factor

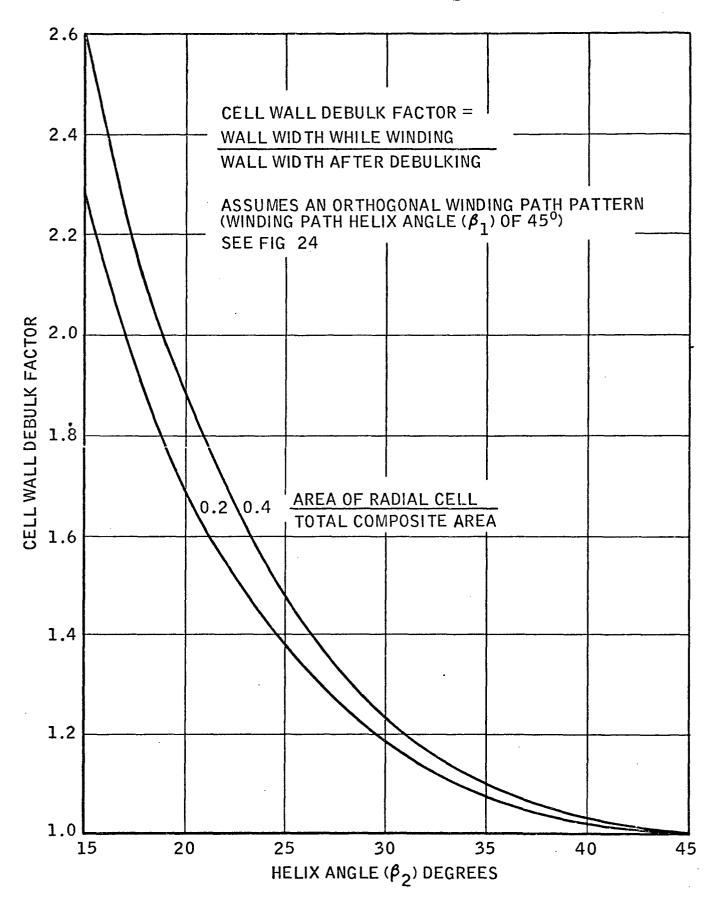


Figure 23
Cell Wall Debulk Factor

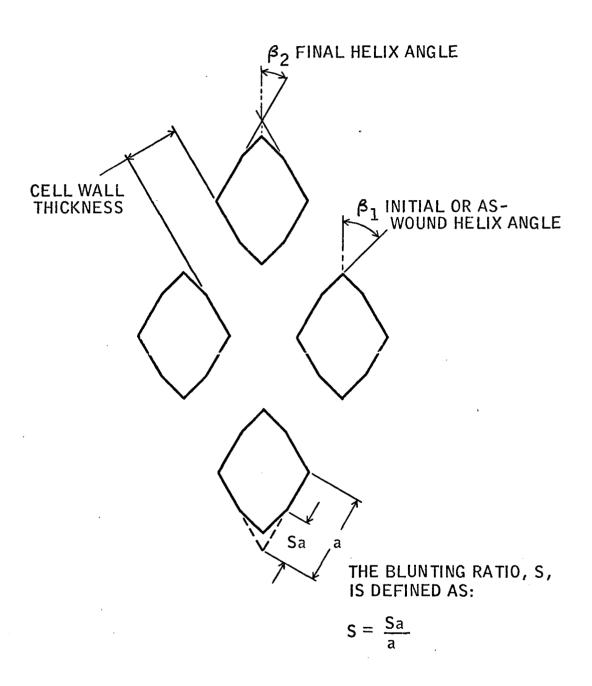


Figure 24

Geometric Parameters of Blunted Cells

TABLE 6

LATERAL DEBULK PROCESS PARAMETERS

Debulk Temperature (°F)		160			
Bag Pressure (psig)		. 30			
	Cylinder	Helix No. l	Helix No. 2	Helix No. 3	
	Time (hr)	9	6	16	
Pre-Debulk Over Stage	Temp (°F)	180 <u>+</u> 5	180 <u>+</u> 5	150 <u>+</u> 5	
·	Revolve Every	First 3 hr-5 min Thereafter-15 min	Same No,]	15 min.	
	Baste Every	First 3 hr-5 min	Same No. 1	15 min.	

Control of resin content during pre-debulk oven staging is difficult. Heating causes the resin to flow out of the part. Basting is an attempt to replace this run-off. The present process incorporates no method of measuring the net resin content change from this step. One method might be to install a rotating spit mechanism in the oven to turn the cylinder fast enough so that it would be self-basting, i.e., loose no resin. Alternatively, a series of cylinder weights could be determined during oven staging. Based on the cylinder weight just before staging, the basting and turning could be regulated to produce zero resin loss. Of the two solutions, the first seems the most practical.

The weighings mentioned in Paragraph a., above were used to estimate cylinder resin content after lateral debulk (Appendix C). This was found to be about 42%, on the average.

c. Scale-Up

Lateral debulk of a 57-in. O.D. flange requires appropriate increases in the dimensions of all lateral debulk fixture components. The components must also be able to handle a part which is cone-shaped on both its ID and OD or one in which at least one surface, ID or OD, is cone-shaped.

The primitive design state of some of the parts of the present fixture should be rectified as a matter of course during scale-up.

7. Cure and Joint Construction

In the normal course of Intremold processing, helically wound cylinders would be cured immediately after lateral debulk, and while still under pressure in the debulk fixture, and orthogonally wound cylinders immediately after filament winding. However, the normal processing sequence was interrupted for four out of the six cylinders in order to construct Intremold III-to-AGCarb transition joints before cure. In the remaining two cases these joints were made after Intremold III cure because of the inability of the uncured Intremold III to withstand the 200 psig AGCarb cure pressure. The construction of each individual joint is discussed in detail in Section III.E. This sub-section will be confined to a discussion of the curing process used on each of the six cylinders.

All of the cylinders, except Helical cylinder number 3 (H-3), and Orthogonal cylinder number 3 (0-3), were cured on male mandrels in an autoclave using Nitrogen at 200 psig, maximum. The cure temperature cycle used for all the cylinders, except 0-3 and H-3, is shown in Table 7. Cylinder (0-3) was cured on a male mandrel in an oven at atmospheric pressure using the temperature cycle given in Table 24 of sub-section IV.F. For reasons discussed in sub-section IV.E. H-3 was cured in a female mold under a Nitrogen pressure of 30 psig. The temperature cycle used is given in Table 21, sub-section IV.E.

-23-

All cylinders, except 0-3, were prepared for cure using a standard technique. This technique is given in detail in the process instructions of Enclosure (1) to Reference (9) dealing with the cure of each cylinder.

a. Inspections

Cylinders were visually inspected after cure, weighed, and all dimensions recorded.

b. Problems

The Intremold III portion of the cylinders which were cured after joint manufacture was damaged by the radial compressive load caused by the maximum cure pressure shown in Table 7. The principal features of this damage were tilting of the radial cells and distortion of the filament windings. This pressure was required to adequately debulk the AGCarb laminated fibrous graphite material used in the joints. A full discussion of this problem and the attempts to find a solution to it is presented in Section IV.A.

c. Scale-Up

Cure of an engine-sized part, in the case, at least, of a helical flange would require a female mandrel on the order of 57 inches inside diameter, plus 0.3 inches to allow for machining. This is based on the conclusion reached in sub-section III.E.lb. that the flange should be cured before making the joint and in Section III.E.5.c that helical cylinders should be pressurized from the inside out during cure. The effect of inside out molding on

TABLE 7

CURE CYCLE FOR CYLINDERS H-1, H-2, O-1, AND O-2

TEMPERATURE (°F)	PRESSURE (PSIG)	INSTRUCTION
RT	30	-
105-130	50	~
130-155	100	₹
155-170	200	~
170-180	200	Hold 20-30 min.*
310	200	Hold 3 hours

^{*} H-1 hold 30 min. 200 - 225°F instead of $170^{\circ}-180^{\circ}F$.

orthogonal cylinders has not been established. If they are adaptable to this type of molding, then a female mold can be used as for the helical cylinders. If not, they should be cured at atmospheric pressure on a male mandrel. Based on Section III.E.5.c, the diameter of this mandrel must be about 0.3 inches less than the final machined inside diameter of the flange at every point to allow for surface clean-up of the Intremold III.

8. Miscellaneous Tooling

Figures 25 and 26 show some of the small tooling referred to in the preceding sub-sections. The functions of the tools shown, except for the male mandrel, have already been described.

The male mandrel was used for lay-up of the joints and for cure of cylinders H-1, H-2, O-1, O-2 and O-3. Grafoil was used as a mold release.

The resin casting and machining fixture was never used for casting of the resin backing. It was found sufficient simply to apply masking tape to the foam mandrel OD while it was still in the cell insertion machine and then spin cast the Flexane.

Not shown in Figure 26 are the adapters used to hold the foam mandrel in the cell insertion machine and those used to hold the pin array in the filament winding machine.

The rubber cutting tools shown in Figure 25 were based on the design given in Reference (2).

9. Process Summary Chart

Appendix F is a detailed process chart summarizing the process parameters and methods which would be used to build helical and orthogonal cylinders based on the experience reported herein. Possible, but unproven, process improvements are not included. In the beginning somewhat different methods and parameters were used which caused deficiencies. Correcting these deficiencies lead to the process described in Appendix F.

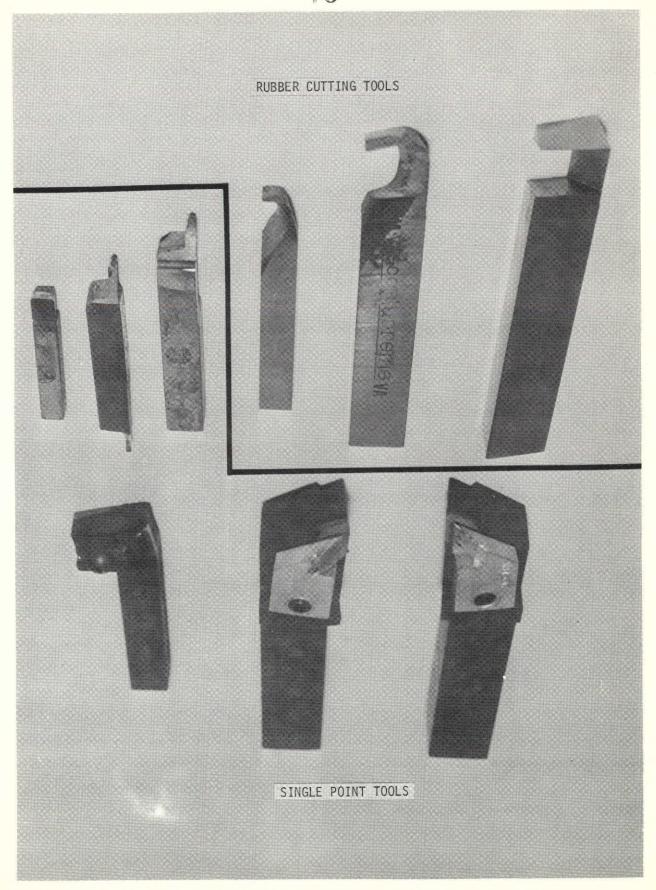


Figure 25. Machining Tools Used

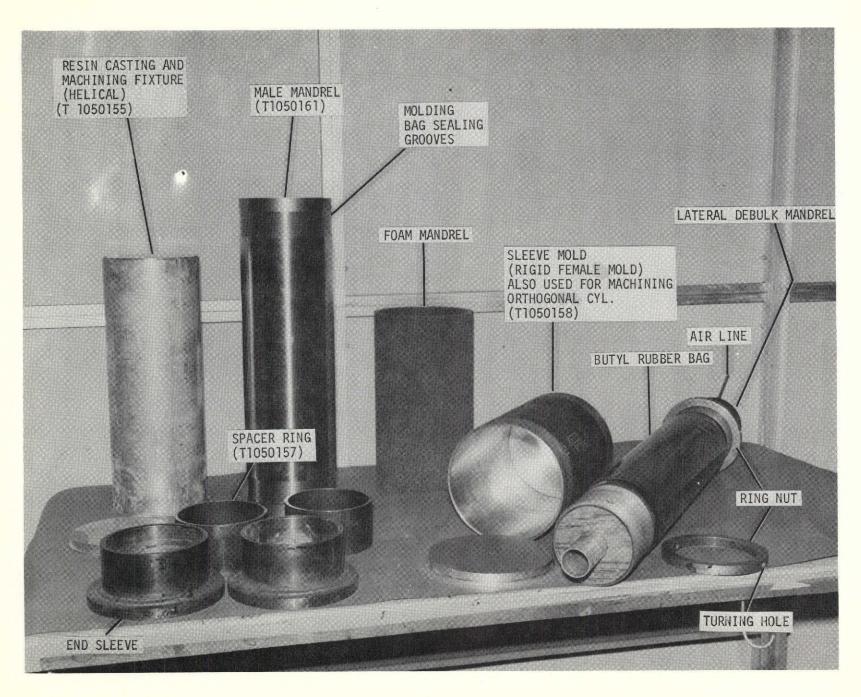


Figure 26. Miscellaneous Tooling



B. MATERIAL OF CONSTRUCTION

General

The Nozzle Extension (NE) forward flange is the potential area of application for Intremold III. During NERVA engine operation, the flange is loaded by the combined action of internal pressure, thrust, steady-state (or transient) temperature gradients, and mechanical loads resulting from the method of attachment to the nozzle aft flange. The most severe loads result from the different thermal expansions of the forward flange made of fibrous graphite and the stainless steel aft flange of the liquid cooled nozzle.

Preliminary finite element stress calculations indicated that. the ability of the prime candidate material (AGCarb), rigidly attached to the aft flange of the primary nozzle, to withstand the loading in the forward flange with sufficient margin to meet both the positive margin of safety and the reliability allocation was doubtful. This was the impetus behind the investigation of Intremold III.

The materials to be selected were the yarn for the radial cells and filament windings, the resin to be used in making the cells and impregnating the filament windings, and the densification impregnant. (The densification impregnant is used to build up bulk density and strength after first cure). The remainder of the materials used were standard to the Intremold III process.

2. Yarns

Union Carbide graphite yarn type WYB 125-1/5 (see Table 8 for yarn code explanation) was chosen for the radial cells and filament windings. Aerojet Liquid Rocket Company, which had been retained to build the Intremold III cylinders, had already investigated several different graphite yarns for use in the Intremold III process. These yarns had been processed into cured cell stock and tested to failure under a tensile load. The data which resulted are presented in Table 8.

Besides high strength, it is important for the composite structure to have a low modulus of elasticity. This is to minimize the stresses

TABLE 8

ALRC STRAND TEST DATA

			YARN		
Strand Test Parameters	WYB 85 1/2	WYB 125 1/5	HMG 15	HMG 50	Thornel 75
Young's Modulus (x 10 ⁶ psi)	1.8	1.8	3	6.6	10
Ultimate Tensile Strength (x 10 ³ psi)	40	67	70*	110	195
		WYB	НМС	ì	Thornel
Yarn Code	1/5 = 5 p1 125 = Dern Code Dernier = -	h nd yarn in WY serie ies/yarn ier(<u>length</u> ier(unit weight)	G = Gra Yai 50 = Yai	dulus aphite	75 = Yarn Modulus x 10 ⁶ psi

^{*} Estimate

caused by thermal expansion. WYB 125-1/5 has the highest strength-to-modulus ratio of the yarns presented in Table 8, and together with WYB 85-1/2, the lowest strand modulus. The strongest fiber, Thornel 75, while providing about twice the strength has about five times the modulus of WYB 125-1/5. Reactor nuclear radiation could be expected to increase this modulus by 20-30%. (Reference (1)) In addition, the rayon precursor for WYB 125-1/5 is the same as that for the UCC grade WCA graphite cloth used to manufacture AGCarb, Thus, a kind of root commonality with AGCarb could be established which would eliminate a variable from consideration.

3. Resins

Since resin selection is independent of composite geometry, (Reference (10)) U.S. Polymeric's resin system F566K, USP 39 Phenolic resin with filler, was chosen for radial cell collimation and filament winding impregnation. System F566K is the leading candidate pre-preg resin for AGCarb, primarily because of its excellent shrinkage characteristics. This choice also provided a good chance for commonality with AGCarb and also a chance to eliminate questions of shrinkage compatibility between Intremold III and the AGCarb portion of the transition joints.

4. <u>Densification Impregnant</u>

Again, the selection of a densification impregnant is independent of geometry. Densification of the cylinders was therefore to be accomplished with the impregnant which was to be selected for AGCarb at the conclusion of screening studies which were then underway. (Subsequently, Allied Chemical Company's 15 V pitch diluted with indine was selected for the cylinders. This selection is documented in Reference (13)).

C. SELECTION OF COMPOSITE GEOMETRY

1. General

The design of Intremold III cylinders requires selection of values for the following quantities:

a. Helical Cylinders

- (1) Radial Cell Configuration
 - a Blunting ratio, S (Figure 24)
 - **b** Initial helix angle, β_1 (Figure 24)
 - \underline{c} Final helix angle, β_2 (Figure 24)
 - d Transverse area
 - e Length
- (2) Area Ratio, r_A (Figure 20)
- (3) Cylinder Dimensions
 - a After debulk
 - b Before debulk,
- (4) Length at Each Reserved for Joint
- b. Orthogonal Cylinders
 - (1) Radial Cell Configuration
 - a Transverse area
 - b Length
 - (2) Area Ratio
 - (3) Cylinder Dimensions
 - (4) Length at Each End Reserved for Joint
 - (5) Amount of Corner Blunting.

2. Cylinder Dimensions

The cylinders were sized for compatibility with existing tooling and to meet budget restrictions. The dimensions which resulted from these

constraints are shown in Table 9. The length reserved for a joint at each end was sized with the possible loading of the entire circumferential joint in mind. It was also assumed that the fill fibers of the AGCarb would lie in the axial direction (this was to parallel the current NE design philosophy) and, for conservatism, that the joint would be a simple lap shear type. An approximate calculation based on these assumptions yielded the reserved length shown in Table 9. This calculation appears in Appendix B.

3. Radial Cell Configuration

For the diamond cells, blunting ratio and initial and final helix angles were chosen to take advantage of existing cell molding and sizing tooling and to distribute more strength in the circumferential than in the axial direction. This distribution trade-off is illustrated theoretically in Figure 27, which was taken from Reference (6).

The transverse cell areas for diamond and square cells which had been developed by ALRC for use with Intremold III were used in the cylinders made for this project. Again, this provided compatibility with existing cell molds.

Cell length had also been standardized for the 0.5 inch cylinder wall thickness for diamond and square cells. This was established as follows (all dimensions in inches):

Final wall thickness	0,50
Machining allowance O.D.	.05
Machining allowance I.D.	.05
Resin backing allowance	,15
Total length	.75

The amount of corner blunting selected for the square cells was the standardized amount then in use by ALRC. Again, this allowed use of existing sizing dies.

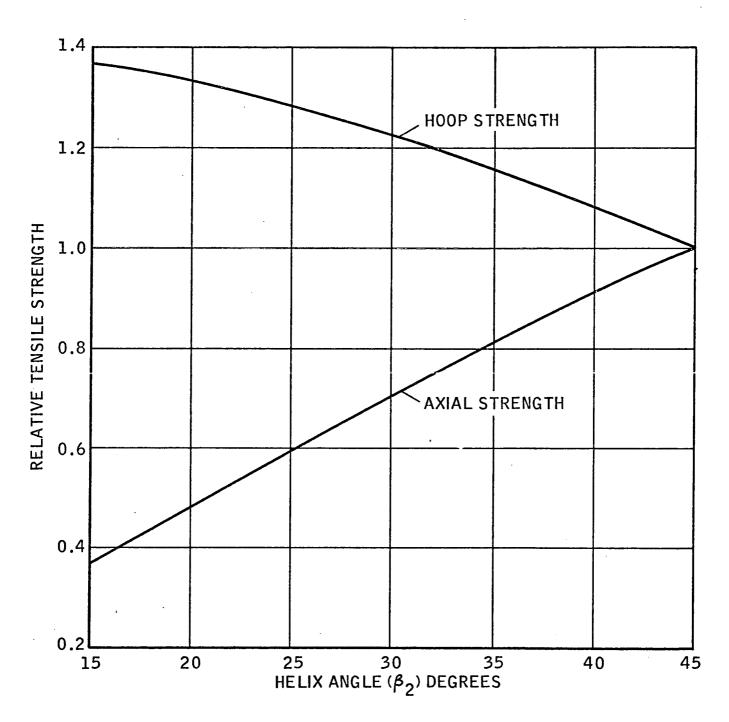


Figure 27
Relative Hoop and Axial Strengths

TABLE 9
FINAL MACHINED INTREMOLD CYLINDER DIMENSION

Length of Intremold (in.)	12
Inside diameter (in.)	8
Outside diameter (in.)	9
Reserved for joint at each end (in.)	3

The cell dimensions selected, as described above, are presented in Table 10. References (11) and (12) for diamond and square cells, respectively, document the cell dimensions selected.

Parameter	Diamond Cell	Square <u>Cell</u>
β	45°	•
^β 2	30°	e
S	.5	See Ref. (12)
A_T	,0016 in, ²	.003 in. ²
L	.75 in.	.75 in.

TABLE 10

RADIAL CELL PARAMETERS

Having accepted some geometries for tooling compatibility does not imply, as far as it is known, that materials properties were compromised.

4. Area Ratio

Decreasing the area ratio increases strength in the θ -z (cylindrical) plane by increasing the fibers available to carry the loads in that plane. Figure 28, taken from Reference (14) and adapted, illustrates theoretically this affect for helical material.

Strength in the direction lying between reinforcing fiber directions will be less than in these directions, with the minimum strength on the bisector of the angle between the reinforcements. However, the modulus in this direction is also lower. These inferences have been drawn from unclassified

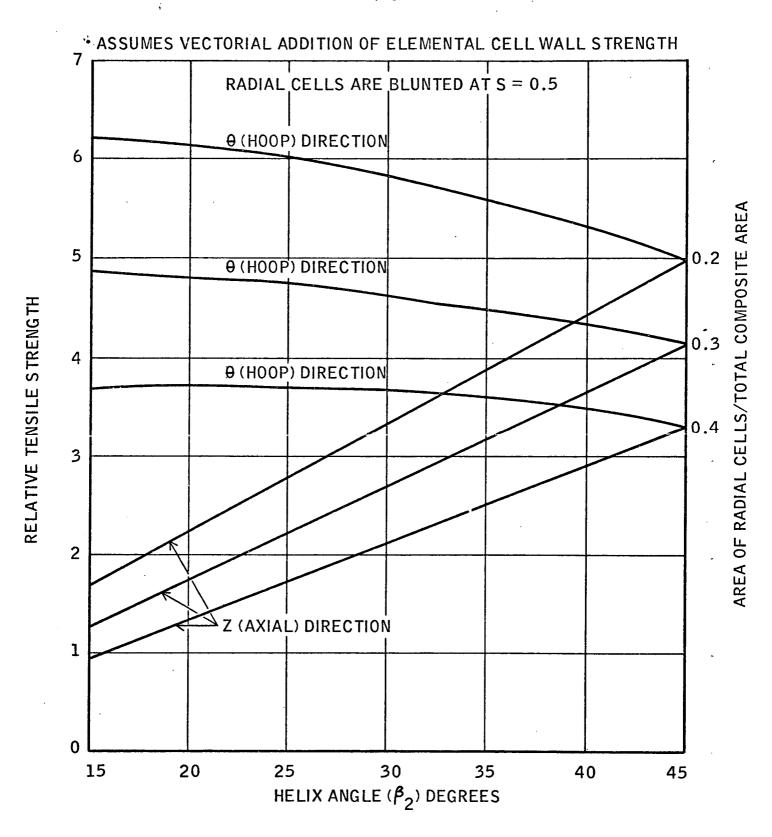


Figure 28
Hoop and Axial Tensile Strengths

-57-

data presented in Reference (7). This data was for flat panels of graphitized orthogonal material with an area ratio about twice that of the cylinders will for this project. The magnitude of the strength reduction along the bisector in these panels was probably increased by the edge effects generated from the necessity of cutting the reinforcing fibers along both edges of the specimens loaded along the bisector. The magnitude of the modulus decrease may also have been increased by the edge effects. The lower bounds for area ratio are the minimum levels of radial strength and θ -z plane shear strength desired. An r_A of 20% was selected based upon the recommendation of ALRC.

5. Helical Cylinder Diameter and Length Before Lateral Debulk

More precise calculation of the shape change factors (see Appendix A) displayed in Figure 21 yields, for the hoop extension factor (f_{θ}) and the axial contraction factor (f_{τ})

$$f_{\theta} = 1.225$$

$$f_{7} = 0.707$$

If the pre-debulked diameter is d_0 and the pre-debulked length is l_0 ,

$$d = f_{\theta} d_{o}$$
 (1)

$$1 = f_z l_0 \tag{2}$$

Substituting from Table 9 for 1 and d (and adding 0.1 in. to d for machining allowance) and solving for $\mathbf{d_0}$ and $\mathbf{l_0}$ yields

$$d_0 = 7.44 \text{ in.}$$

$$1_0 = 17.02 \text{ in.}$$

The required I.D. is found by subtracting two times 0.6 in. (.75 - flexane allowance) from d_0 .

$$d_{o_{\text{ID}}} = 6.24 \text{ in.}$$

. -6₽3×

D. JOINT DESIGN

General

With the selection of Intremold III as a forward flange candidate material came the necessity of joining the Intremold III flange to the AGCarb nozzle extension liner. There are two types of joints possible: strictly mechanical, such as a bolted flange, or "integrated", i.e., one which is made during processing of the liner and flange, and which, at least in part, is bonded together by the resin matrix. This report deals exclusively with the integrated joints. Twelve integrated joints were built, two on each cylinder, utilizing the three inches reserved at the ends of each cylinder.

a. Design Method

In order to avoid pitfalls, all joints were first conceptually designed and laid out to engine-sized scale. Steady state stress plots (no transient analyses available) from finite element solutions using AGCarb were studied to gain a qualitative understanding of the stress distribution in the flange. It was assumed that the stress distribution for Intremold III would be similar and joint components were therefore located with reference to this distribution. For example, the bond lines of all joints were located as close as possible to the plane of zero-shear. After this step, the manufacturing process required for each engine-sized concept was explored for feasibility. The advice of manufacturing and materials personnel was sought to determine what was possible. If the concept remained attractive after these steps had been taken it was scaled down to the half-inch thickness and 9 inch OD of the cylinders. During scale down the objective was to preserve the essential geometry of the concept.

Scaling down from a 54-inch ID, 1.5 inch thick flange with a general conical shape to an 8 inch ID, 0.5 inch thick cylinder generates problems. The slope of the cone can be used to increase the joint strength under tensile loads. Obviously, this can not be done on a cylinder. The thinner, constant thickness cross-section of the cylinder forced the modification of some features of most of the concepts. Since each joint was unique, it was clear that

specimens would have to be cut from them for testing, rather than trying to pull the entire joint. Wrapping hoop windings on the joint OD, a feature of most engine-sized concepts, was therefore useless and was dropped from the small scale versions (with one exception (see III.D.l.d.). Finally, the smaller diameter made manufacture more difficult.

Twenty-one integrated joint concepts were devised.

Thirteen concepts were selected for final design. This step consisted of sizing the parts of the joint using approximate calculations (see Appendix B) and assumed material properties, specifying dimension tolerances, adding the design to the design drawing of the selected cylinder, and writing instructions for making the joint. Of the 13 joints selected only ten different joints were actually installed on the cylinders. The reasons for the two duplicate joints are given in sub-sections III.D.1 and III.D.6. The leading assumptions used in cylinder joint design are listed in Table 11.

b. General Description

The integrated joints are of two fundamental types: lap or butt. One straight lap joint was included as a baseline as well as to determine its feasibility. All of the other lap joints are modified straight laps. Low radial tensile strength and low axial shear strength are the principal weaknesses of lap joints. Hoop winding the engine-size joints was the main means selected to improve resistance to radial tensile loads. Resistance to axial loads (which are transmitted as axial shear in the bond area) was increased by arranging the load path so that part or all of the shear at the joint interface would be transmitted directly to the composite structures, bypassing the graphitized resin bond, and by increasing the shear area.

The axial tensile strength of a bonded butt joint is the joint's weakest feature. Shear strength in the plane of the bond line is also low. In the case of the one butt joint built for this project an axial clamp bridging the joint was utilized to carry the axial tensile loads. In the paragraphs which follow the particular application of these general ideas to each joint is explained in more detail.



TABLE 11

JOINT DESIGN ASSUMPTIONS

Bond Shear Strength			1000 psi
AGCarb Warp Tensile Str	rength		5000 psi
AGCarb Warp Compression	Strength		9500 psi
Intremold Cross-pin She	ear Strength	>	>9500 psi
Intremold Axial Compres	ssion Strength	:	>9500 psi
Loads are Uniformly Dis	stributed		
Intremold Axial Tensile	strength		
Helical		and there are	4000 psi
Orthogonal			8000 psi

TABLE 12

CYLINDER JOINTS

NO.	NAME	<u>CYL INDER</u>	CLASS
I	Comb Tooth	Helix 1*	Divided Load Path Lap
I(M)	<pre>Comb Tooth (Modified)</pre>	Ortho 3	Same
11	Straight Lap	Helix 1	Simple Lap Shear
II(H <u>)</u>	Wound Lap	Helix l	Simple Lap (Hoop Wound)
III	Interleave	Ortho 3*	Augmented Shear Area Lap
٧	Ledge Lap	Helix 2	Divided Load Path Lap
A11.	Washboard Lap	Ortho 1	Same
VIII	Thread Lock	Helix 2	Same
XVI	Wave Lap	Ortho 1 & 3	Same
XVIII	Bayonet	Ortho 2	Augmented Shear Area Lap
XIX	Interlock	Ortho 2	Divided Load Path Lap
XX	Butt Clamp	Helix 3	Modified Butt
VXIV	Buck Tooth	Helix 3	Divided Load Path Lap

^{*}Initial assignment

Because of the unwound cells this joint is most easily made with an AGCarb shingle lay-up. Each ply must be pre-drilled and laid up individually over the pins. This would be very difficult to do with rosette plies because of the angle they make with the Intremold surface. With the continuous wind tape-wrapping process it would be nearly impossible.

The details of the fabrication of the comb tooth joint are given in sub-section III.E.6.b.

b. Straight Lap

Figure 30 shows this joint in cross-section. The

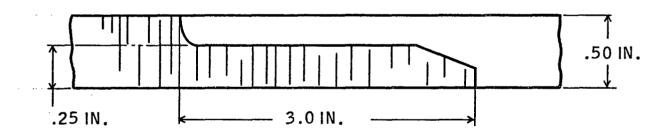


Figure 30 Straight Lap Joint

strength of this joint depends primarily upon the strength of the graphitized resin matrix between the AGCarb and the Intremold. Some direct load pick-up by the radial cells is possible since the joint was made with both constituent composits B-staged. Thus, some penetration of the lower lamina by the cells in the joint area when the materials become plastic under cure conditions is possible.

The simplicity of this joint makes it very attractive from the manufacturing and financial points of view. It can be used easily with any of the four liner lay-up candidates, shingle, rosette, 3-D woven fabric, or tape wrap. On a scale of increasing manufacturing difficulty (and therefore cost), the straight lap joint is the easiest to make, and the comb tooth the hardest.

The hoop-wound straight lap (the only exception to the no-hoop-winding rule) was made to investigate the affect of processing on hoop windings applied before cylinder cure. Otherwise it does not differ from the straight lap joint.

c. Interleave

Figure 31 depicts the interleave joint, Because

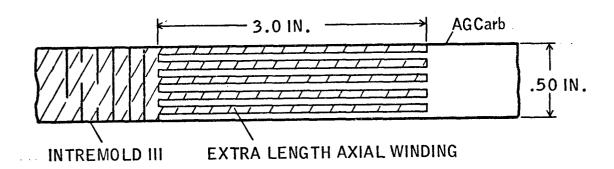


Figure 31
Interleave Joint

construction of this joint required that the Intremold filament windings be extended three inches beyond the end of the Intremold III cylinder, and then sandwiched between alternating layers of laminated material ("interleaved"), it is applicable,

practically, only to orthogonal material and shingle lap liner lay-ups. The axial windings of the orthogonal composite are easily extended beyond the cylinder during filament winding. Since the curvature of the plies of the shingle lap lay-up has a constant radius, they can be completely inserted between the filament layers. This cannot be done with other lay-ups.

The interleave joint attempts to increase the shear area, and thus reduce the shear stress on each AGCarb-to-Intremold bond, by stacking alternating layers of axial filaments and laminate plys. In theory, given the low ratio of wall thickness to inside diameter and laminate and filament layers of equal thickness, the shear area should increase by a factor of about 2(n-1), where n is the number of axial filament layers, over the straight lap joint (Appendix B). This assumes all the filaments will remain intact and can be kept straight and uniformly distributed about the circumference of the cylinder. Practical experience showed these to be poor assumptions.

The reduction in bond shear stress of this joint probably would be at the expense of axial tensile strength. The axial filaments would be robbed of the load sharing ability of the other members of the composite—matrix, cells and circumferential windings.

The joint was never completed because of difficulties with the exposed axial filaments. These difficulties are discussed in detail in sub-section III.E.6.b.

d. Ledge Lap

This joint is shown in Figure 32.

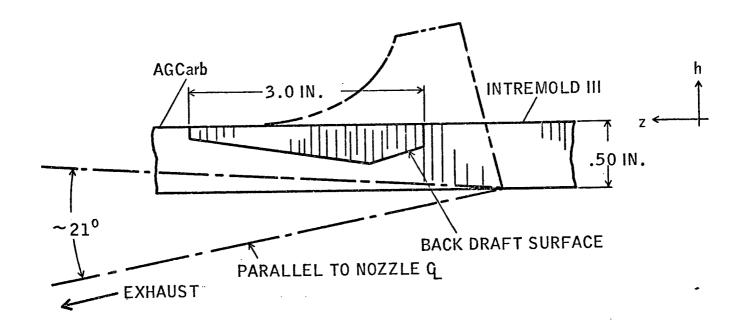


Figure 32 Ledge Lap Joint

The AGCarb portion of this joint was made and machined separately as a "pre-form". During cure, the Intremold III portion was molded on to it. The back draft surface provides a lock between the liner and the flange which is independent of the resin bond. To give a clearer understanding of this joint the full scale concept has been drawn in Figure 32 (phantom lines). The geometry of the back draft surface was sized using approximate calculation to prevent its flattening under the maximum steady-state radial compression stress estimated at its location. Shear loading of the back draft surface was minimized by locating it near to the zero shear axis (as described in sub-section a.). The shear strength of the liner was improved by making it with a "rosette" lay-up so that the r-z plane shear is not interlaminar (as it would be with a shingle lay-up) but tends to load the reinforcing fabric fill direction fibers in compression (in the case of the flange; in the wall of the rosette cylinder liners the warp fibers were run in the axial direction to help prevent tensile failures

in the joint specimens outside of the joint area. The warp tensile strength of AGCarb is about twice its fill tensile strength).

e. Washboard Lap and Wave Lap

Figure 33 shows both of these joints. These two joints explore ways of loading the pins directly without introducing sharp corners (as

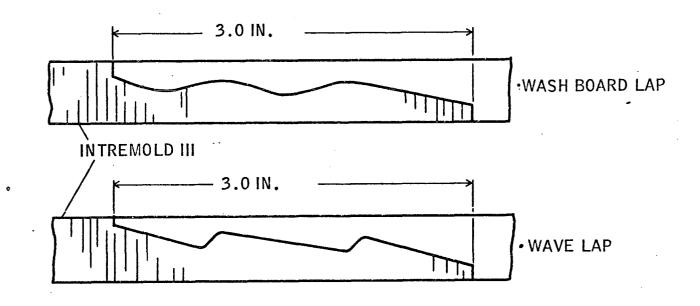
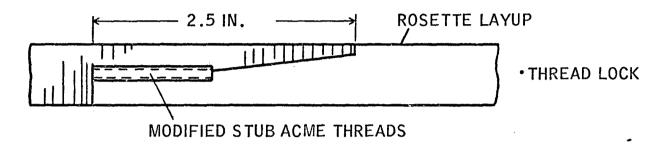


Figure 33
Washboard Lap and Wave Lap

do the joints of sub-section h, for example). They can be used, because of the absence of corners, with a shingle lap liner. However, the probability of damaging wrinkles is greater than with the straight lap joint. A rosette liner lay-up was employed on both joints to minimize wrinkling and to maximize shear strength in the liner.

In the engine-sized concept, the hoop windings would keep the liner material clamped to the joints' contours.

f. Thread Lock, Interlock and Buck Tooth



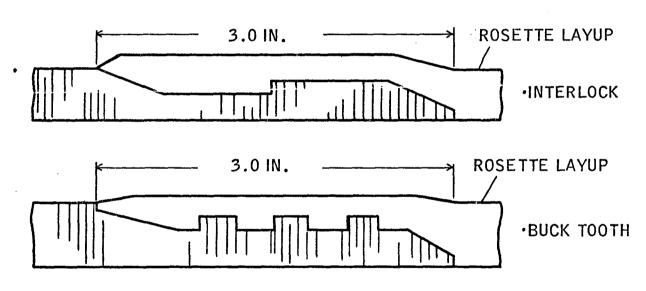


Figure 34

Thread Lock, Interlock and Buck Tooth

A.

The three joints illustrated above represent different approaches to the same goal: transferring the joint shear load directly to composites to by-pass the low shear strength of the matrix. Buck Tooth grew from a desire to utilize a threaded joint concept but to avoid the manufacturing steps of internal thread machining of the Intremold and pre-form making.

The rosette lay-up was utilized in the liners of all three joints. This is required in joints of this type because of the sharp radial transition of the joint contours and to provide sufficient shear strength in, for example, the threads of the thread lock joint. Figure 35 illustrates how the rosette increases shear strength.

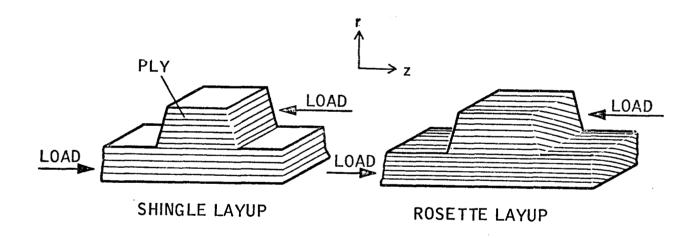


Figure 35
Shingle and Rosette Lay-up Shear Load Path

Note that in the right hand drawing the load distributed over the thread face is transmitted by the ply fibers into the cylinder. In the left hand drawing the load must be transmitted through the relatively weak interlaminar bond between plies.

g. Bayonet

Figure 36 is an illustration of the Bayonet Joint.

The shear area of this joint is a little more than double that of the straight lap

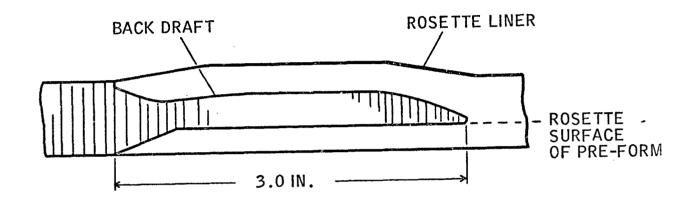


Figure 36
Bayonet Joint

joint. The joint also incorporates back draft on the upper surface of the Intremold III bayonet to help the bayonet resist slipping out of the liner "scabbard" under axial tensile loads.

In order to simulate building the joint on the conical NE mandrel surface the liner was made in two parts. The lower part of it was laid-up separately and machined for a close sliding fit and placed underneath the bayonet. Then the upper half was laid up on the upper bayonet surface and the upper pre-form surface

h. Butt Clamp

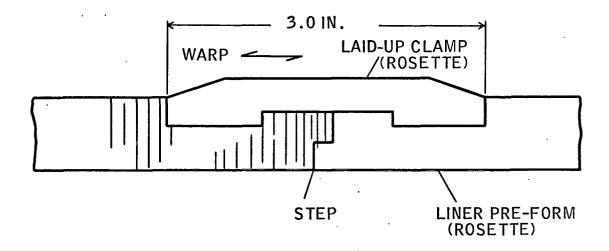


Figure 37
Butt Clamp

Because of the low tensile strength of the matrix bond the axial tensile strength of a butt joint is low. Two modifications to a simple butt joint were added to improve this characteristic. The first is the clamp. The clamp has the strong warp fibers running axially and was made with a rosette lay-up. Under tensile loads the clamp tends to develop a moment about the butt joint as shown below for a joint without the butt step.



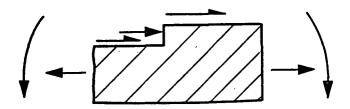


Figure 38
Butt Joint Tensile Load Free Body Diagram

The step, shown above in Figure 37, helps the butt joint resist peeling apart under the influence of this moment. Instead of resisting the peeling action with the tensile strength of the resin bond alone the peeling load shared is carried in compression and shear at the step face.

The most important advantage of the butt joint is that axial compressive loads are transmitted directly between joint halves with the bond in compression, rather than in shear as in the straight lap shear joint. Both the joined materials have high compressive strength compared to the bond shear strength. Here again the use of the rosette lay-up is dictated by the various ledges in the joint contour.

3. Rosette Ply Patterns

Two methods were employed to calculate the shapes and number of plies of the various joint rosette ply patterns. The first method used will be called the "geometric" method and the second (and preferred) method the "analytical" method. Figure 39 illustrates the geometric method.

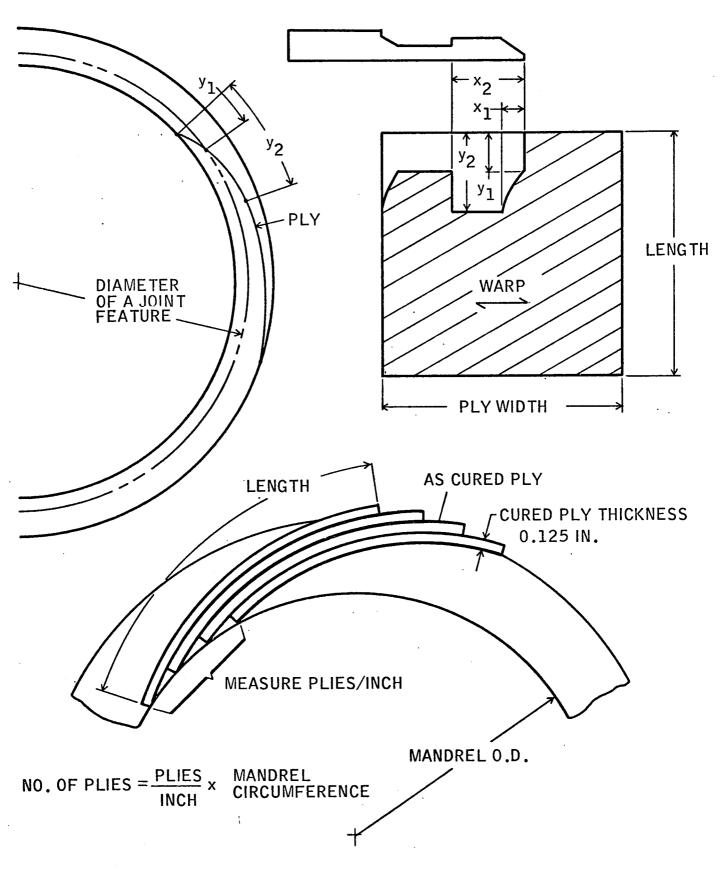


Figure 39
Geometric Method

In both methods the ply length and cured thickness must be known.

a. Geometric Method

As Figure 39 illustrates, the geometric method determines the coordinates of each joint feature by laying the ply out on a cross-section of the cylinder and plotting the intersection of the diameter of each joint feature with the ply. The distance along the ply length to each intersection is picked off and transferred to a layout of the ply. Combined with the axial coordinate of the feature these coordinates described the ply shape required. The number of plies is determined by a 10/1 blow-up showing a cured cylinder cross-section. The plies are drawn in and the number of plies/inch on the ID measured. Since the ID is known the total number of plies may now be computed.

This technique has two inherent errors. First, small plotting errors on the cylinder cross-section can cause large errors in the Y coordinates of the ply contour. This is because of the small angle the plies make with the ID and OD surfaces of the cylinders. But, most importantly, the curve of the ply must be assumed, or approximated by trial and error. The ply contour error this introduces can be significant. The consequences of the errors in this technique are discussed in sub-section III.E.3.b.

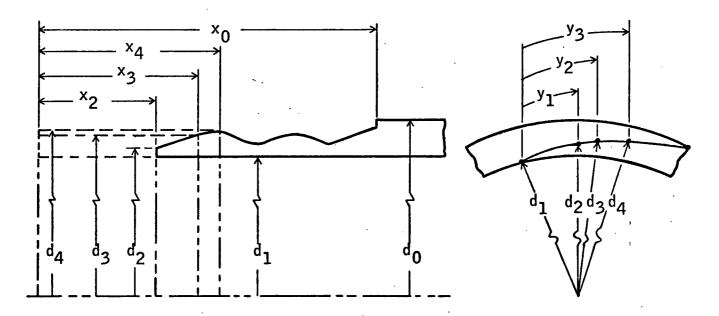


Figure 40 Analytic Method

b. Analytic Method

The analytic method does not require knowledge of the ply curve. The joint contour is intersected by the outer surfaces of a series of imaginary cylindrical annular rings of inner diameter $\mathbf{d_1}$, and outer diameter equal to the desired feature diameter. At $\mathbf{d_0}$ each ply has a cross-sectional area equal to

$$A_{p} = 1 t \tag{3}$$

where

l = ply length, and t = ply cured thickness (.0125 in.). The cylindrical annulus with diameters d_0 and d_1 , has cross-sectional area

$$A_{1} = \frac{\pi}{4} \left(d_{0}^{2} - d_{1}^{2} \right) \tag{4}$$

The number of plies required is therefore

$$N_{p} = \frac{A_{1}}{A_{p}} = \frac{\pi}{4} \left(\frac{d_{0}^{2} - d_{1}^{2}}{1 t} \right)$$
 (5)

The plies/inch on the mandrel is

$$n_{p} = \frac{N_{p}}{\pi d_{1}} \tag{6}$$

where $n_p = plies/inch$.

For small t/l ratios the cross-sectional area of the i th imaginary annulus is

$$A_i = \frac{\pi}{4} (d_i^2 - d_1^2) = N_p y_i t$$
 (7)

and therefore,

$$y_i = \frac{\pi}{4} \left(\frac{d_i^2 - d_1^2}{N_p t} \right)$$
 (8)

This method gave excellent results.

4. Qualitative Comparison of Joints

Tables 28 and 29, Appendix E, rank the joints under three criteria: estimate of strength, ease of manufacture, and cost. These ranks are subjective and stem from the design and manufacturing experience with each joint. Using this method the best all around joint is Interlock, and the worst, Comb Tooth.

IV. MANUFACTURING HISTORY

A, HELICALLY WOUND CYLINDER NO. 3

1. <u>Design Description</u>

Helix 1 was the first cylinder to be built. As finally configured, its design is recorded on ANSC drawing 1139309. (Appendix C). The joints assigned to Helix 1 are listed in Table 12. Originally, the Comb tooth joint (Type I) was assigned to the end of Helix 1 now occupied by the Wound Lap Joint (II (H)). For reasons discussed below, the Wound Lap Joint was substituted for Comb tooth after lateral debulk.

2. <u>Manufacturing Narrative</u>

The radial cell stock for Helix 1 had the characteristics recorded in Table 12 below.

TABLE 12 A
HELIX 1 CELL DATA

Process Identification No. (PIN)	Collimation Run	<u>Mold</u>	Width Across Flats (in.)	Density $\frac{gm}{cc}$
200]	.3	104	,039 - ,043	1,20
	3	105	Not Recorded	1.29
	4	106	.039041	1.31
	4	107	.041043	1.30*
	4	108	.038043	1.34
	4	109	.038042	1.29
	5	110	.037041	1.30
	. 5	111	.038041	1.30
	5	112	.037041	1.32
	5	113	.038042	1.29
	2	114	.036039	1.31

^{*} Single Specimen

Cell manufacture was completed as described in section III.A.3. Several problems and some deviations from the procedures of section III occurred thereafter.

After cells had been inserted in about the first three axial inches of the foam mandrel, the ratchet mechanism and locking pawl operating and locking the indexing gear malfunctioned. This caused about one circumferential row of cells to be inserted with incorrect inter-cell spacing and incorrect cell row-to-cell row spacing. Because of the space taken up by the incorrectly placed cell holes, the row could not be re-punched. In order to save the time already expended, the 3-inch long portion of the foam mandrel which had been laid up with cells before the casualty was cut off and processed separately. This 3-inch long cylinder was designated "Mod. 2" (denoting its expected lateral debulked length) and the remaining 14-inch portion, "Mod. 10". (This casualty is documented on IR 512310, Appendix B).

After separation of Mod. 2 from its parent cylinder, resin backing was applied to it and cured and the foam mandrel was dissolved with MEK.

While the Mod. 2 cylinder was being processed further, the Mod. 10 cylinder was completing cell insertion. For this particular cylinder the final step in this process was to counter-sink most of the cells in the combtooth joint area. (The reason for counter-sinking is given in sub-section III.C.1.c.). The depth of counter-sinking was 0.3 in. from the foam mandrel outer surface. Figure 41 illustrates this process step.

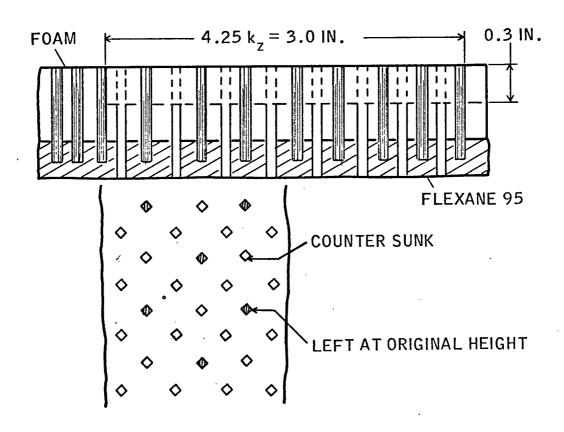


Figure 41 Comb Tooth Joint Cell Counter Sinking

After the cells were counter-sunk, the resin backing was cast and cured and the foam mandrel was dissolved (a minimum of three rinses in methylethyl ketone requiring a total time of about eight minutes).

Filament winding of the Mod. 10 cylinder was begun first. The filament winding procedure deviated from that described in sub-section III.A.5. in order to wind the combtooth joint area correctly. Windings were applied until the

tops of the counter-sunk cells were reached. However, at this point the axial length of the counter-sunk area was discovered to be about an inch too short. This error was corrected by grinding 0.3 in. off the proper pins until the required joint length (shown in Figure 41) was obtained. Then, filament winding was continued on the portion of the cylinder outside of the joint area until the tops of the cells were nearly reached. (Winding to the tops of the cells is not done because the windings tend to ride up over the cell tops).

Five days after beginning filament winding of Mod. 10, filament winding of Mod. 2 began using an auxilliary wooden fixture. This was done so that filament winding of the Mod. 2 and Mod. 10 cylinders could be completed at the same time and the cylinders could be processed together after filament winding and save time.

Post-winding impregnation and pre-lateral debulk oven staging of Mod's 2 and 10 were conducted simultaneously on both cylinders without incident.

Prior to lateral debulk, the possibility of the 0.3-inch thick cylinder wall in the combtooth joint area being too thin to withstand the internal pressure load imposed by the butyl rubber bag was raised. There was also the possibility that the expanding metal female mold bearing against the tops of the pins would provide enough support to prevent damage. In the event, lateral debulking seriously changed both the Mod. 2 and Mod. 10 cylinders. The damage was caused by poor mold release during debulk, and the inability of the combtooth area wall thickness to withstand bag pressure.

The mold release agent used was polyethylene film applied to the interior surface of the rigid female mold. The filament windings stuck to this film. When the cylinders were forced from the mold the last layer of filament windings was pulled up.

Insufficient resin polymerization allowed the center portion of cylinders Mod's 2 and 10 to retreat from their molded diameter, yielding an hourglass shape (this is referred to as "memory"). This damage is shown in Figure 42 and Figure 43.

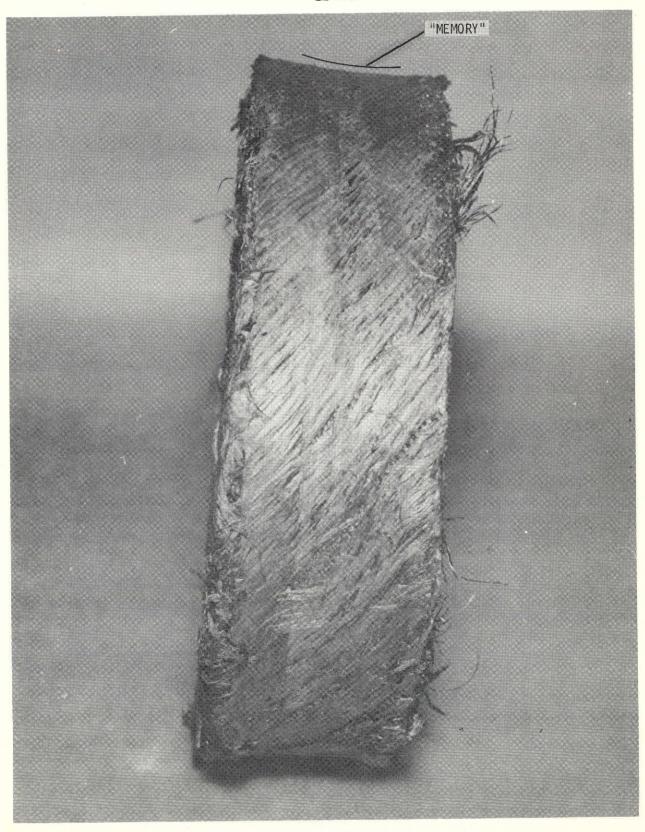


Figure 42. Cylinder Mod 2 After Lateral Debulk

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Figure 43 also shows the damaged comb tooth area. Indicated on the figure is the "ballooning" caused by the comb tooth area wall failure. The ballooning resulted in widespread radial cell displacement and misalignment.

The deep resin puddles which formed in the comb tooth area were an un-anticipated defect. These were probably caused by resin draining from the main part of the Mod. 10 cylinder into the low point represented by the comb tooth area. This resin then formed puddles because it was prevented from escaping by the end sleeves and the molds.

Inspection of the comb tooth area of Helix 1, Mod. 10, showed that this area was too badly damaged to repair. Both Mod. 2 and Mod. 10 were placed on a male mandrel. Mylar shrink tape was applied to the cylinders to draw them to the mandrel surface. Then a vacuum bag was placed over them and evacuated and the cylinders were heated in an oven at 180°F for two hours at atmospheric pressure. The combination of heat softening and atmospheric pressure restored the parts to their cylindrical shape. However, the cell tilting resulted in a cylinder wall thickness less than 0.6 in. and a cylinder OD less than 9.1 in. (This casualty is documented in IR 512438A, Reference D).

After cooling, the comb tooth area was machined away and replaced by a straight lap joint. Plans were made to circumferentially filament wind this joint before cure to observe the affect of subsequent processing on the filament windings. These observations would provide clues to help select the best processing for filament windings.

Grafoil was selected to replace the polyethylene film mold release for all future molding operations.

The shingle lap lay-up had been selected for the straight lap joint. Joint construction was to take place before cure (and after lateral debulk, in the case of helical cylinders) to maximize the strength of the bond between the joint halves. A geometrical method was used to determine the ply

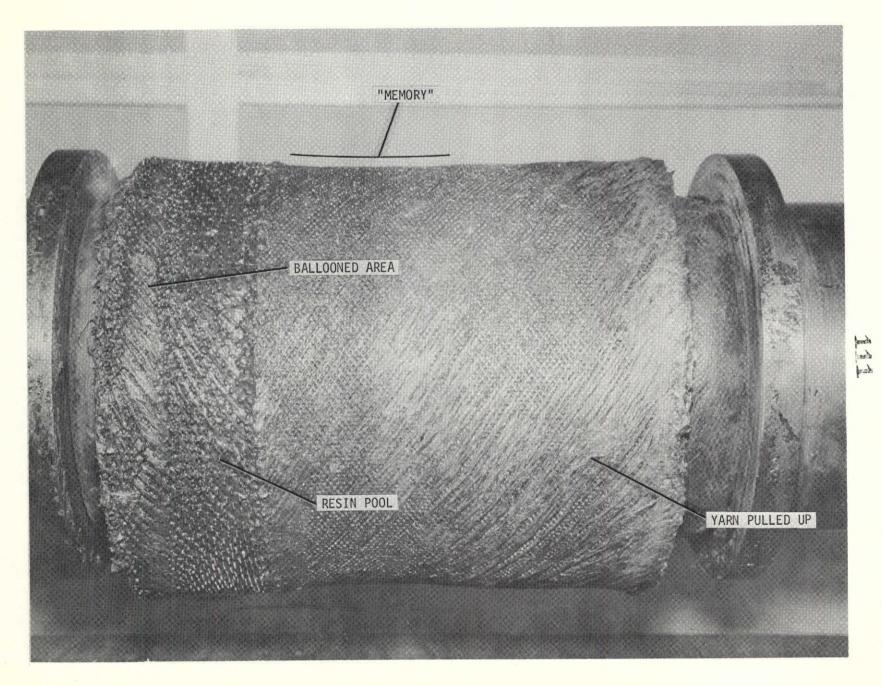


Figure 43. Cylinder Mod 10 After Lateral Debulk

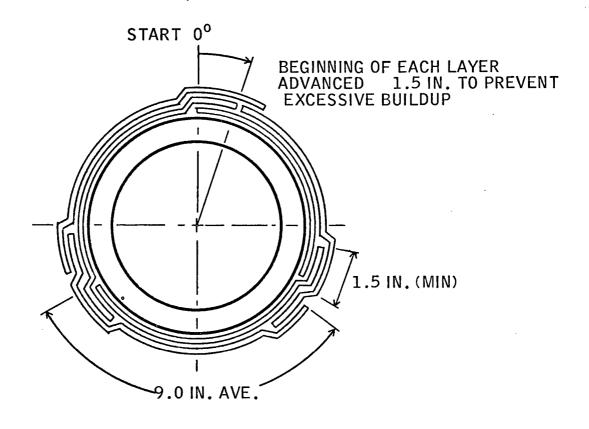
dimensions and the quantity of plies required. A 10:1 scale cross-section of the joint was laid out and each ply drawn in using its debulked thickness (0.0125 in.), and ignoring overlaps. The number of plies was then determined. The ply circumferential width was specified as averaging nine inches, somewhat arbitrarily. The considerations in choosing ply width are (Reference (10)):

- a. Long plies reduce ply cutting time and increase hoop tensile strength, but tend to produce more wrinkles when debulked. Wrinkles reduce strength (Reference (3)).
- b. Short plies tend to reduce wrinkles and increase cutting time, but reduce hoop tensile strength because of the increased number of overlaps required.

Ply overlap was chosen as 1.5 inches, minimum, to give sufficient interlaminar shear strength to the overlap joints (Reference (10)). The warp fibers of the plies were oriented parallel to the cylinder axis to maximize the axial tensile strength in the AGCarb half of the joint. This helps to ensure against failure in the AGCarb, rather than in the joint, when testing the joint. Layup design data for the Type II joint is summarized in Table 13. Figure 44 shows the shingle lap lay-up schematically.

Minimization of lay-up wrinkling is of great importance, particularly in shingle lap lay-ups. In addition to choosing the correct ply circumferential length, ply pre-debulking and debulking during layup help to control wrinkles. Pre-debulking is done by compressing the cut plies between the heated platens of a press, which changes their thickness from about 0.025 in, to about 0.0175 in. This helps to prevent wrinkles by reducing the layup thickness buildup between debulks.

For Helix 1, Mod. 10, in-process debulking took two forms. The first method tried was to heat mylar shinrk-tape which had been wrapped around the layup. Heating the tape causes it to contract and compress the plies. This method is fast, but not very effective. Eventually it was abandoned in favor of autoclave debulking. Preparation of the cylinder for debulking is the same as for



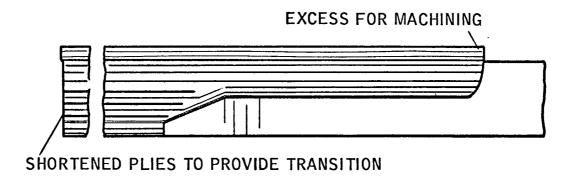


Figure 44
Shingle Lap Lay-Up

. TABLE 13
STRAIGHT LAP LAY-UP DATA

	Type II Joint
Total Number of Plies	224
Circumferential Ply Width (in.)	8.5, 9.0, 9.5, depending upon radial location.
Axial Ply Length (in.)	6.0, 3.03 - 3.89, depending upon radial location.
Overlap (in.)	1.5 (minimum)

cure and is described in Reference (9). During debulking, the cylinder was heated to 180°F and remained at this temperature for one hour under 200 psig nitrogen pressure. This temperature was high enough to cause the resin to flow, and, therefore, to allow the layup to be compressed. However, the time at temperature was too short and the temperature was not high enough to cause significant polymerization of the resin. This was a standard debulk cycle.

For Helix 1, Mod. 10, a total of 28 in-process debulks were performed: 26 with mylar shrink tape and two autoclave debulks (performed last). Autoclave debulking of Mod. 10 revealed a serious problem: The Intremold III material in the uncured state could not withstand the 200 psig pressure required to debulk the AGCarb. The radial compressive load generated tilted all the pins about 20° in the circumferential direction. This in turn caused distortion of the filament windings. Axial pressure exerted on the Intremold III by the AGCarb layups at its ends caused it to hump and wrinkle,

Three solutions were chosen for investigation. The first, and initially the preferred solution (because of the desire to maximize bond strength) was to "super-stage" the resin in subsequent cylinders before joint construction. By "super-staging" is meant prolonged low-temperature oven heating (180°F or below) to polymerize the resin to the point where it can support the cells under 200 psig loading, but still cross-link with the resin in the AGCarb half of the joint. This method was tried with little success on Ortho 1 (because of the design of the ledge lap joint, Helix 2 was processed in the old way) and with moderate success on Ortho 2. The difficulty with super-staging was that it was hard to judge when the cylinders had been staged enough, and no time or extra material was available for experimentation. (This problem is documented in IR 512438A).

A second method considered was to debulk and cure at some pressure, less than 200 psig, which could be tolerated by the Intremold III. This procedure was anticipated to be of doubtful value because the amount of AGCarb debulk depends on pressure.

The third method was to cure the cylinders before joint construction. This would provide a rigid structure which would be able to withstand 200 psig. The question which remained to be answered, however, was: What will the affect be on joint strength? The implementation of this method was therefore held in abeyance.

Two sets of AGCarb lap shear joint specimens were prepared to investigate methods two and three. One set of joint specimens was cured with one of the adherents already cured before making the joint. The other set with both adherents "green" (uncured) before curing the joint. Specimens in each set were processed at 30, 100 and 200 psig to evaluate the affects of pressure. After cure the joint specimens were subjected to tensile loading until failure and the shear load to fail computed from the joint bond area and the ultimate tensile load.

The results plotted in Figure 45 show that the average reduction in mean strength caused by bonding cured to green material is about 15%. (Neglecting the anomally at 100 psig on the cured-to-uncured shear strength curve). This reduction seems nearly independent of pressure (it is 12% at 200 psig and 18% at 30 psig). The affect of reducing pressure was to reduce the amount of debulk, as expected.

The maximum pressure to which Intremold III had been subjected prior to this program was 30 psig, during lateral debulk. The reduction in AGCarb debulk at this pressure, and even at 100 psig, as indicated by the increased thickness plotted in Figure 45, was judged to be unacceptable.

At the completion of lap shear specimen testing, only Ortho 3 and Helix 3 remained to be built. It was imperative to complete these cylinders essentially undamaged. With the exception of Interleave and its replacement Wave Lap, the designs of the remaining joints did not depend heavily on the resin bond for strength, except under radial tensile loads. (Hoop windings to be installed on engine scale parts would provide strength to resist such loads). Rather than

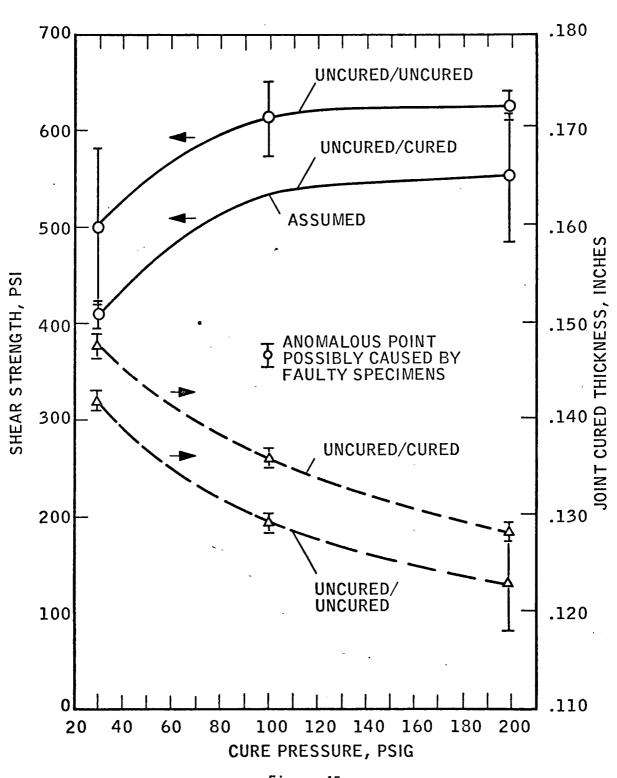


Figure 45
AGCarb Lap Shear Test Data

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design a new joint, Wave Lap was selected to replace Interleave (which was dropped for reasons discussed in sub-section III.E.6.b.) to further evaluate the effect of method three on a joint after completion of all processing through final graphization. For these reasons Helix 3 and Ortho 3 were processed using Method 3.

After the final autoclave debulk of Helix 1, Mod's 2 and 10, the filament windings on the outside of joint Type II (H) on cylinder Helix 1, Mod. 10, were applied. A channel for the winding was prepared by machining the layup over the Type II (H) joint to a diameter of 9.00 in. over a 3.4 in. length. A four-yarn filament using WYB 125-1/5 yarns was wound into this channel to a depth of about 0.180 in. Each layer of yarn was impregnated by hand using F556K resin mix (USP-39 and filler). A total of 199 grams of yarn and 155 grams of resin mix were applied, yielding a resin content of about 39%. This resin content is high, based on AGCarb experience, and may result in delamination, and uniform manual application of the resin was difficult. In addition, the dry yarns tended to fray. Winding with pre-pregged yarn would have prevented both of these problems.

Helix 1, Mod's 2 and 10, were cured on the same mandrel using the cure cycle given in Table 7. Figure 46 shows their condition after cure (Mod. 2 adhered to Mod. 10 during cure).

3. Conclusions

- a. The Comb Tooth joint cannot be used with Helical cylinders.
- b. The debulk and cure pressure used for AGCarb cannot be applied to Intremold III before or during cure.
 - c. The polystyrene film mold release is unsatisfactory,
- d. Joint filament winding with pre-pregged yarn should be investigated.
- e. More staging during lateral debulk is required to prevent "memory" effects.
 - f. Mylar shrink tape layup debulking is unsatisfactory.

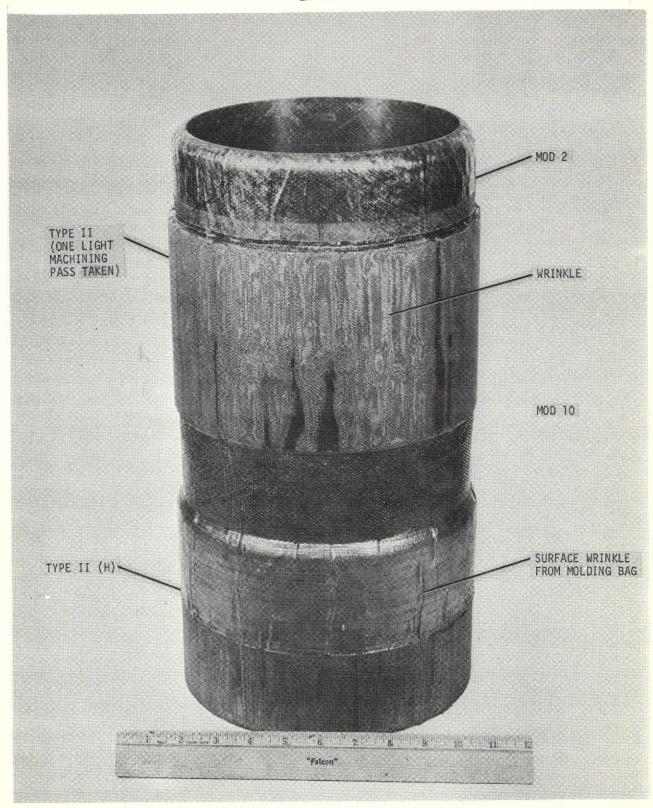


Figure 46. Helix 1, Mod 2 and Mod 10

B. HELICALLY WOUND CYLINDER NO. 2

1. Design Description

The design of Helix 2, the second cylinder to be built, is recorded in ANSC drawing 1139564 (Appendix C). The joints assigned to Helix 2 were Ledge Lap and Thread Lock.

2. Manufacturing Narrative

The radial cell stock for Helix 2 has the characteristics recorded in Table 14, below.

TABLE 14
HELIX 2 CELL DATA

Process Identification No. (PIN)	Collimation Run	<u>Mold</u>	Width Across Flat (in.)	Density (gm/cc) (cured)
2003	1	100	0.032 - 0.040	1,24
	1	101	0,038 - 0,042	1,33
	2	102	0.037 - 0.040	1.35
	2	103 ·	0.038 - 0.041	1,25
	2	104	0.038 - 0.042	1,33
	2	105	0.037 - 0.042	1,29
	3	106	0.038 - 0.041	1,24
	3	107	0.037 - 0.041	1,28
	3	108	0,038 - 0.042	1,25
	3	109	0,038 - 0,042	1,24
	4	110	0.037 - 0.041	1.31
	4	111	0.038 - 0.041	1.31

The manufacture of Helix 2 proceeded without significant deviation from Section III until joint construction.

The design of the Ledge Lap and Thread Lock joints required machining of the Intremold III and AGCarb portions of these joints. Accordingly, two cylindrical layups were made and debulked (using the cycle given in Table 7 through 180°F). The rosette cylinders so formed ("pre-forms") were machined to ANSC drawings 1139573 (for the Ledge Lap joint) and 1139615 (for the Thread Lock joint). The Intremold III portion of Helix 1 was machined to ANSC drawing 1139564-2 configuration. These parts are shown in Figure 47. The machine tool used throughout was the "rubber cutting tool" in Figure 27. The method used to build the pre-forms for Thread Lock and Ledge Lap was the same for each joint. The plies used for both joints were rectangular pieces of US Polymeric FM5228 pre-preg material 10 in. by 4 in., in the warp and fill directions, respectively. After pre-debulking, as previously described for Helix 1, these plies were laid up on the grafoil covered surface of steel mandrel T-1050161 (Figure 26). This surface had previously been marked in one-inch intervals by masking tape. The spacing of the plies on the mandrel had been calculated as 14.5 $\frac{\text{plies}}{\text{in}}$. The plies were placed on the mandrel surface in the rosette configuration with the long (warp) dimension along the cylinder axis and held in place with rubber bands. Using the masking tape markers as quides the spacing between plies was adjusted to give 14 or 15 plies/inch, alternatively. After this, mylar shrink tape was wrapped around the entire lay-up surface and the assembly was heated with a heat gun. The heating and the resultant shrink tape compression caused the plies to compact and stick together. When the assembly had cooled, the tape and rubber bands were removed. After cooling, the lay-up was prepared for debulk in the standard way described in Reference (9) Enclosure (1). The debulk cycle used was the same as the cure cycle of Table 7. but the maximum temperature used was limited to 180°F to prevent excessive advance of the resin. Debulk of FM-5228 laminar lay-ups utilizing these parameters usually achieves about 95% of the maximum debulk obtainable using the full cure cycle. The ten-inch long rosette cylinder and its supporting mandrel were then taken to the machine shop to be cut in half and machined as described in the preceeding paragraph.

Figure 47. Parts of Helix 2 - 1139564-1

The preceding description was given in some detail to show the general procedure followed in making a rosette lay-up. Some of the other layups were not pre-forms, but were laid-up directly on the Intremold III joint contour. In these cases, for rosette lay-ups, no debulk step was required. Once the mylar shrink tape was removed the assembly went directly to cure.

The Helix 2 lay-up parameters are summarized in Table 15.

TABLE 15

LEDGE LAP AND THREAD LOCK JOINT LAYUPS

Number of Plies/Join	370
Dimension of Plies (before lay-up machining)	10 in. (warp <u>)</u> by 4 in. (fill)
Type of Lay-up	Rosette
Ply Stagger on Mandrel Surface	14.5 plies/inch
Warp Orientation	Cylinder axis

Inspection of the 1139564-2 Intremold III cylinder revealed that the internal threads of the Thread Lock joint end had been improperly machined. During remachining, excessive tool run-out caused the threads to advance too far axially into the cylinder. When the pre-forms were mated to form assembly 1139564-1, the AGCarb threaded portion was not long enough to mate with the extra Intremold III threads. As a result, a circumferential crease in the Intremold III surface was formed during cure (Figure 48). (IR 512438B)

Tolerance build-up during machining caused the feather edge observable in Figure 47. Experience with machining this edge on 1139564-2, and

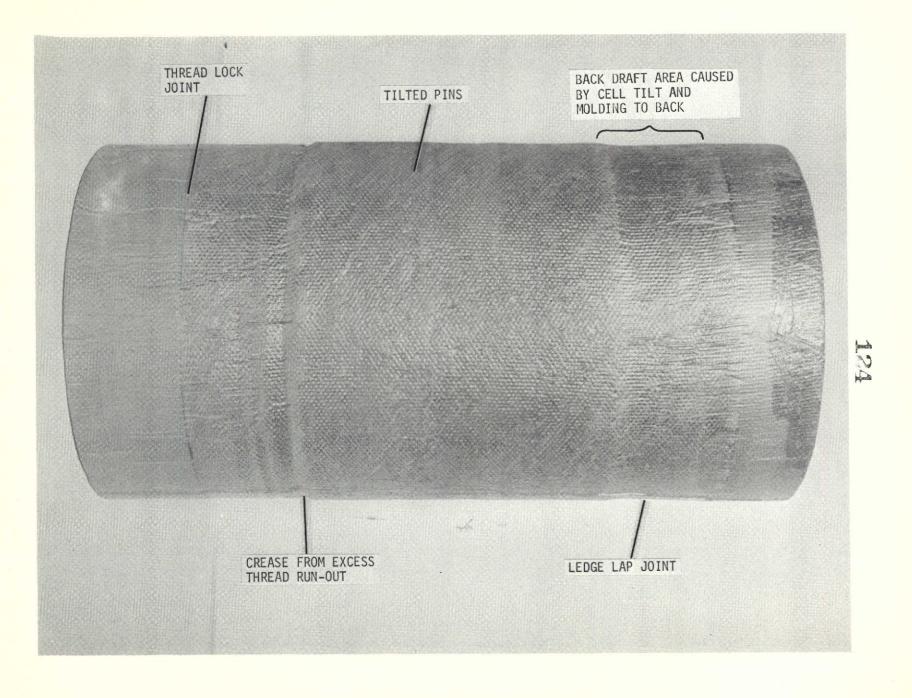


Figure 48. Cured Helix 2 - 1139564-1

edges on other cylinders, indicated that machined edge heights less than about 0.1 inch on Intremold are not feasible.

Cure of Helix 2 followed the temperature and pressure cycle given in Table 7. The radial cells of Helix 2 were tilted, and as for Helix 1, the cell tilt resulted in an undersized OD and wall thickness (this effect accompanied all cases of cell tilt). But the overall damage was less than that inflicted on Helix 1. This was so probably because the rosette lay-ups for the joints of Helix 2 did not require in-process debulking, as did the shingle lap lay-ups of Helix 1.

3. Conclusions

Helix 2 demonstrated that AGCarb and Intremold III could be machined to fairly complex shapes while in the partially polymerized state described above (which is usually referred to as the "B stage". "C stage" is a cured part. "A stage" refers to the non-polymerized resin. The FM5228 pre-preg is B staged when received.) At the beginning of the project doubts had been expressed by Materials and some experienced project personnel as to the feasibility of doing this.

In view of the undesirability of pressurizing uncured Intremold III to 200 psig the Ledge Lap joint concept will be dropped from consideration.

C. ORTHOGONALLY WOUND CYLINDER NO. 1

1. <u>Design Description</u>

The design of Ortho 1, the third Intremold III cylinder to be built, is recorded in ANSC drawing 1139310 (Appendix C). The joints assigned to Ortho 1 were Wave Lap and Washboard Lap.

2. Manufacturing Narrative

The radial cell stock for Ortho 1 has the characteristics recorded in Table 16, below.

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ORTHO 1 RADIAL CELL DATA

Process Identification No. (PIN)	Collimation Run	Mo1d	Width Across Flat (in.)	Cured Density gm/cc
2002	1	100	.050052	1.25
	1	101	.050052	1.31
	2	102	.050052	1.31
	2	103	.050052	1.33
	2	104	.050052	1.32
	2	105	.050053	1,29
	3	106	.050052	1,32
	3	107	.050052	1,29

The manufacture of Ortho 1 proceeded in standard fashion through post-winding impregnation. After this step, the cylinder was superstaged for a total of 15 hours at $150^{\circ} \pm 5^{\circ}$ F. During this period it was revolved every 15 minutes, but basted for only the first three hours.

After cooling, the cylinder was placed on mandrel T-1050161-1, using grafoil as a mold release, and the joint areas were machined to the Wave Lap and Washboard Lap configurations on ANSC drawing 1139310.

Liner ply cutting templates were prepared using the geometric method described in sub-section III.C.l.k. Because of the error in this method, the cutouts in the plies were too deep and insufficient material remained to fill

up the volume in the liner above the joint interface. As a result, the joint contour showed through. This can easily be seen in Figure 49, which shows Ortho l immediately after mylar shrink tape removal.

Also observable in Figure 49 is the typical waviness of the axial filament windings mentioned in sub-section III.A.5.

Table 17 gives the layup design data for Wave Lap and Washboard Lap joints.

TABLE 17

ORTHO 1 JOINT LAYUP DESIGN PARAMETERS

Joint		Wave and Washboard Lap	
No. Plies/Joint	No. Plies/Joint		
Ply Basic Dimensions(in.) Warp		5 6	
Ply Stagger on Mandrel (<mark>pli</mark> in	10.5		
Lay-up		Rosette	

After the joints were laid up the entire assembly was cured using the cycle given in Table 7. Figure 50 is a photograph of Ortho 1 after cure.

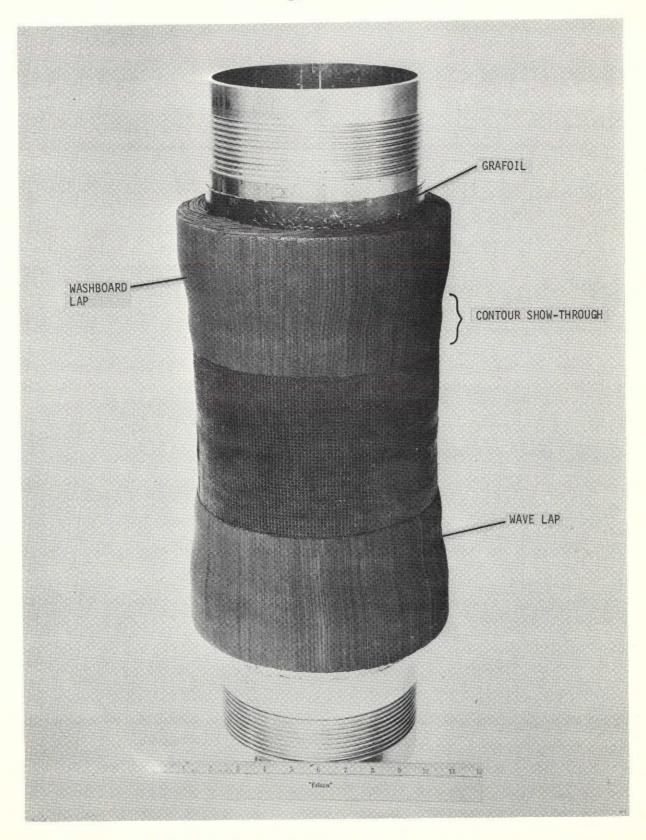


Figure 49. Cylinder Ortho-1 After Joint Lay-Up

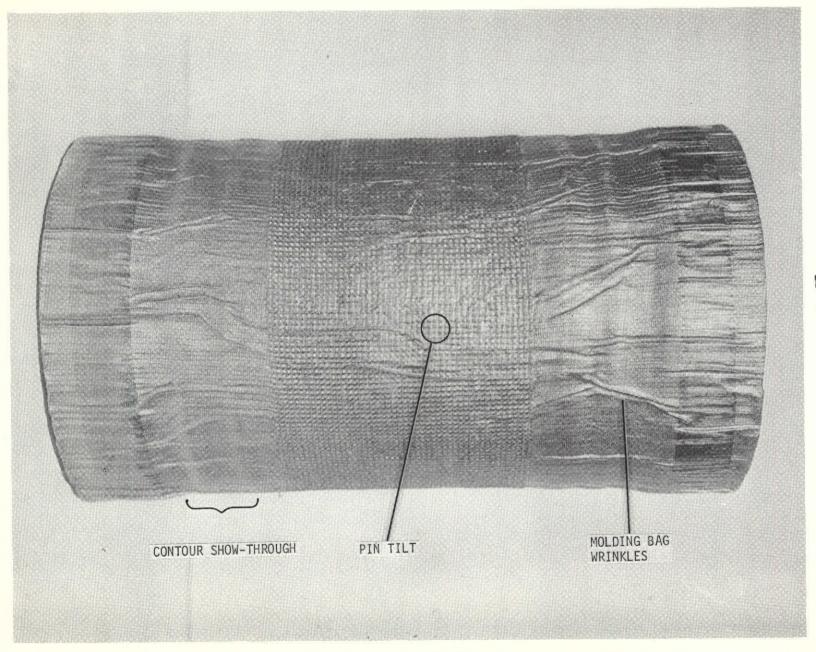


Figure 50. Ortho 1 After Cure



The ridges on the surface are from the molding bag. The joint contour show-through and cell tilt are evident. However, the damage was less than that sustained by Helix 1. The overall appearance of the cylinder was the best up to that point. (see IR 512438D for all deficiencies)

3. <u>Conclusions</u>

Experience with Ortho 1 showed that the error in the geometric method of layup design was unacceptable.

D. ORTHOGONALLY WOUND CYLINDER NO. 2

1. Design Description

The design of Ortho 2, the fourth cylinder to be built, is recorded in ANSC Drawing 1139565 (Appendix C). The joints assigned to this cylinder were Bayonet and Interlock.

2. Manufacturing Narrative

The cell stock for Ortho 2 has the characteristics given in Table 18.

TABLE 18

ORTHO 2 CELL STOCK DATA

Process Identification No. (PIN)	Collimation Run	<u>Mold</u>	Width Across Flat (in.)	Cured Densitygm/cc
2004	1	100	.051053	1.33
•	1	101	.050052	1.30
	2	102	.051052	1,33
	2	103	.050052	1.33
	3	104	.050052	1.30
	3	105	.047052	1.32
	3	106	.050053	1.32



Processing of Ortho 2 was standard through post-winding impregnation. After this step it was super-staged for 16 hours at $150^{\circ} \pm 5^{\circ}$ F. During the first three hours of this period basting was carried out. Upon cooling, it was machined to configuration 1139566-2, shown in Figure 51.

As described in sub-section III.C.l.i, the lower half of the bayonet joint was made as a rosette pre-form and the upper half of the joint laid-up directly on the Intremold III contour and the pre-form after they had been mated. The pre-form was made in the same way as those made for Helix 2. The ply patterns for the upper half of the Bayonet joint and for the Interlock joint were first determined by the geometric method, after applying a correction to the assumed curvature of the ply. It was evident during lay-up that the patterns were still cut too deeply. The analytic method was used to calculate the number of extra plies required to supply the missing volume. The final lay-up design parameters are listed in Table 19. After Ortho 2 the geometrical method was abandoned in favor of the analytical method.

TABLE 19
ORTHO 2 JOINT LAY-UP DATA

Joint		Bayonet Pṛe-form	Bayonet Upper	Interlock
No. of Plies		170	350 "	442
Ply Spacing (plies	:/in.)	6.88	14.2	18.0
Div Pasia Dim (M)	Warp	5.0	5.0	5.0
Ply Basic Dim.(M)	Fill	4.0	4.0	6.0
Lay-up		Rosette	Rosette	Rosette

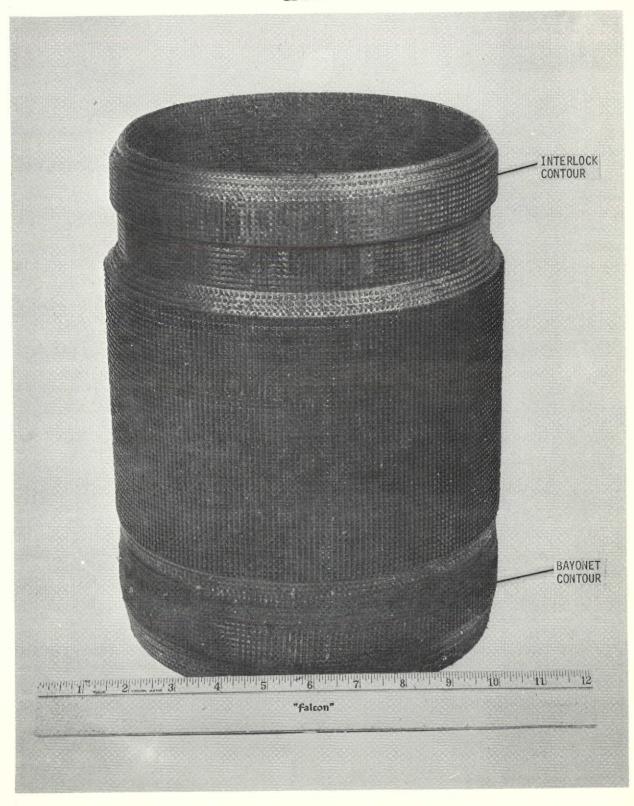


Figure 51. Ortho 2 After Joint Contour Machining

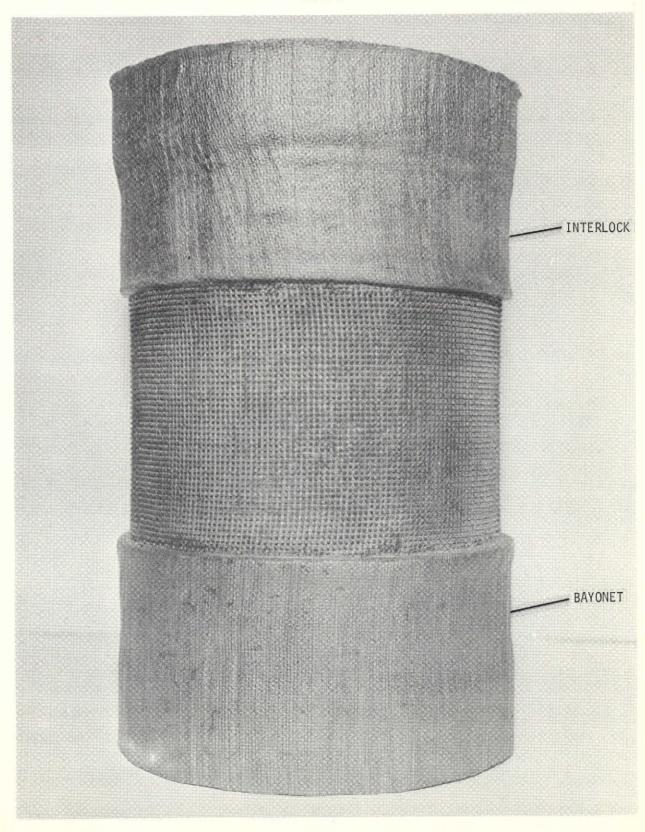


Figure 52. Ortho 2 After Cure

After laying up both joints the assembly was cured. Figure 52 shows Ortho 2 in the cured state. Cell tilt occurred but was minimized. No joint contours and very few bag wrinkles were visible. (IR 512438E)

3. Conclusions

The ability of the super-staging to ameliorate, but not to prevent, cell tilt was evident.

E. HELICALLY WOUND CYLINDER NO. 3

1. Design Description

The design of Helix 3 is recorded in ANSC Drawing 1139583 (Appendix C). The joints assigned were Buck Tooth and Butt Clamp.

2. <u>Manufacturing Narrative</u>

The characteristics of the cell stock used in Helix 3 are shown in Table 20.

TABLE 20 HELIX 3 CELL DATA

Process Identification No. (PIN)	Collimation Run	Mold	Width Across Flat (in.)	Cured Densitygm/cc
2005	1.	100	.037042	1.25
	1 .	101	.038042	1,27
	2	102	.041043	1,29
	2	103	.039042	1.22
	2	104	.039041	1.24
	2	105	.038041	1.23
	3	106	.039042	1.35
	3	107	.039042	1.32
	3	108	.039042	1,34
	3	109	.038042	1.33
	4	110	.037041	1,28
	4	111	.038041	1,30

135

Processing of Helix 3 followed the general Intremold III process for Helical cylinders given earlier up through removal of the expanding female mold during lateral debulk. Inspection of the cylinder after this process step revealed that it had not reached its proper dimensions and shape. Its shape was skewed, and one end had contacted the mold, but the other had not. This condition probably was caused by human error in placing and adjusting the banding clamps which control the expansion of the expanding metal mold. One end of the mold was allowed to expand too much and the butyl rubber bag "bottomed" at this end.

The shape and dimensions of the cylinder were successfully corrected by placing it in a female mold, preparing it as for cure, and heating it to 180°F for 20 min. under 30 psig nitrogen pressure. Then it was removed from the mold and inspected. The decision having already been made to cure the cylinder prior to joint manufacture, it was thereupon placed in a female mold again and cured using the cycle in Table 21. Low pressure and a female mold were employed to insure that no tilting of the cells resulted and to tension and straighten the filament windings.

TABLE 21

CURE CYCLE FOR HELIX 3

Temperature (°F)	Nitrogen Pressure (psig)	Hold
RT	30	
105 - 130	· 30	
130 - 155	30	
155 - 170	30	
170 - 180	30	
300 - 310	30	3 hrs after 300°F

During resin backing removal prior to lateral debulk the cylinder had been centered in the machining fixture using three rings of tape. The pressure from the tape had indented the outer surface slightly while the inner surface had been machined smooth. During subsequent molding operations in female molds, the outer surface had become cylindrical, and the ring indentations had been transferred to the inner surface. It was therefore necessary to machine the cylinder inside surface to obtain cylindricity. The net result was that the inside diameter of Helix 3 became 8.161 inches, instead of 8.00 inches. (IR 512438C)

The mold used to restore the cylinder's proper shape and dimensions stuck to the cylinder despite the grafoil mold release used. The mold was cut away from the cylinder. This was the last remaining female mold of the proper inside diameter. Its destruction forced the use of a mold with an inside diameter of 9.2 inches. A machining pass to clean up the outside surface of Helix 3 after cure resulted in a final outside diameter of 9.085 inches. The resulting wall thickness was 0.46 inches. (IR 512438C)

After cure Helix 3 was machined to the 1139583A-2 configuration shown in Figure 53. Also shown in Figure 53 is the rosette pre-form made for the Butt Clamp joint. This pre-form (1139583A-3) was made in the same general way as the pre-forms for Helix 2 and Ortho 1. Note the uniform, wrinklefree filament winding produced by the "inside-out" molding.

The plies for the buck tooth and butt clamp joints were laid out using the analytic method. The lay-up design characteristics for both joints are given in Table 22.

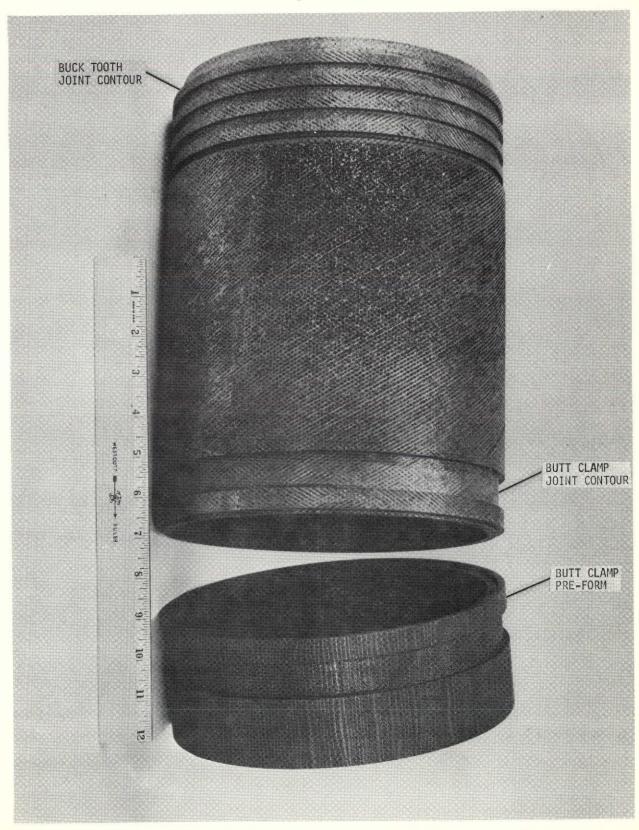


Figure 53. Helix 3 After Cylinder Cure With Butt Clamp Pre-Form



: TABLE 22
HELIX 3 JOINT LAY-UP DATA

Joint		Butt Clamp Pre-Form	C1amp	Buck Tooth
No. Plies/Joint	267		262	319
Ply Basic Dim.(in.)	Warp Fill	3.75 6.00	2.90 6.00	5.00 6.00
Ply Spacing (Plies)	10.23		9,3	12.45
Lay-up		Rosette	Rosette	Rosette

The joints were laid-up and cured using standard procedures with excellent results (no photograph available).

3. Conclusions

The uniform condition of Helix 3 after cure showed the superiority of inside-out molding for the Intremold III material.

The non-standard diameter of Helix 3 resulting from surface clean-up (and previous experience with some of the other cylinders) showed that the 0.05 in. machining allowance on the inside and outside wall surfaces was insufficient. A good conservative allowance would be 0.15 in. on each wall, or 0.30 in. on each diameter. This extra cell length will increase the cost of the cells and of filament winding over that figured for the 0.1 in. diameter allowance.

Grafoil is an adequate, but not infallible, mold release. More work needs to be done to improve mold release performance.

F. ORTHOGONALLY WOUND CYLINDER NO. 3

1. Design Description

The design of this cylinder is recorded in ANSC Drawing 1139566B (Appendix C). The joints finally assigned were Comb Tooth (modified) and Wave Lap (originally Interleave).

2. <u>Manufacturing Narrative</u>

The characteristics of the radial cell stock used in Ortho 3 are shown in Table 23.

TABLE 23
ORTHO 3 CELL STOCK

Process Identification No. (PIN)	Collimation Run	<u>Mold</u>	Width Across Flat (in.)	Cured Density gm/cc
2006	1	101	,050 - ,052	1,34
	2	102	.048052	1,34
	2	103	.048052	1,33
	2	104	.050052	1,33
	2	105	.050052	1,33
	.3	106	.048053	1,33
	3	107	.050053	1.37

The manufacture of Ortho 3 was standard up to cell insertion. The final step in this process, as for Helix 1, was the counter-sinking of the proper cells in the comb tooth area. As described earlier all the cells in the last 0.5 inch of the comb tooth area were counter sunk to allow more room to wind and anchor the axial windings in the main body of the cylinder. Figure 54 shows the counter sink pattern.

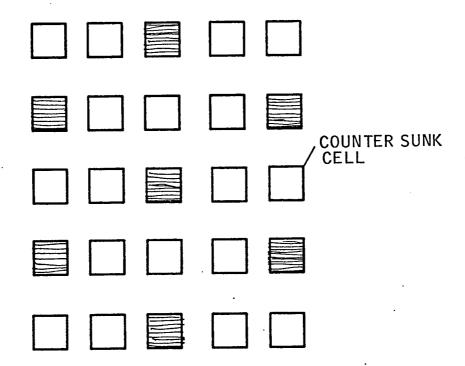


Figure 54
Ortho 3 Comb Tooth Countersinking

Also as described earlier, during filament winding the axial filaments for the Interleave joint were extended three inches from the end of the cylinder. Elaborate precautions were taken to keep these integration yarns dry during subsequent impregnations and oven staging. A great deal of time was spent washing resin from them with alcohol. More precautions were taken to protect them from damage during resin backing removal and removal from the machining fixture and transfer to a male mandrel for joint lay-up. All this proved of no avail, as will be described.

Filament winding was completed with the comb tooth area wound to a wall thickness of 0.3 in., as for Helix 1.

After post winding impregnation the cylinder was superstaged at 150°F for 24 hours and at 180°F for 8 hours. This was done in an attempt to reduce the resin flow during cure to minimize the wetting, and therefore embrittlement, of the integration yarns.



Ortho 3 was the last orthogonal cylinder. While the use of low-pressure inside-out molding during cure was very attractive, in view of the good results obtained with Helix 3, this procedure was rejected. The risk, though probably small, of breaking any of the circumferential filament windings could not be taken. Instead, Ortho 3 was given an elaborate atmospheric pressure cure in an oven. The cycle for this process is shown in Figure 24. A "U" shaped foil channel perforated on the cylinder main-body side and on the bottom was fitted into the completely counter-sunk filament anchoring area to catch resin trying to "pool" in the comb tooth area.

Table 24
ORTHO 3 CURE CYCLE

Temp. (°F)	Duration (hr.)	<u>Instruction</u>
Ramp to 150	1 (min.)	
150	3	Revolve every hour
180	6.	
190	12	Open oven and inspect if still tacky and gooey
200	12	
215	- 12	·
230	12	Check for foaming on way to 250. If present hold at 230.
250	12	at 250,
300-310	2	

The reason for the extended cycle was to insure against boiling of the liquid volatiles which come off the cylinder as the resin cures. Boiling would sprinkle the cylinder with voids.

After cure, despite all precautions, the interleave yarns were soaked and brittle. The yarns were removed and the Wave Lap joint substituted for the Interleave joint,

Construction of this Wave Lap joint was identical to the Wave Lap joint on Ortho 1, except that the analytical method was used for determining the lay-up design. The lay-up design parameters are given in Table 25.

TABLE 25
WAVE LAP JOINT LAY-UP

Joint		Wave Lap
No. of Plies		266
Ply Basic Dim. (in.)	Warp	3.5*
Fry basic Dim. (in.)	rim. (in.) Fill	6,0
Ply Spacing (Plies)		10.67
Lay-up		Rosette

^{*} Shortened to save pre-preg.

The shingle lap lay-up for the Comb Tooth (modified) joint was designed by utilizing a 10:1 scale drawing of the joint, as for Helix 1. The design parameters for this lay-up are shown in Table 26.

as follows:

TABLE 26

ORTHOGONAL 3 COMB TOOTH JOINT LAY-UP DATA

	//·//
Total Number of Plies	264
Warp Orientation	Cylinder Axis
Circumferential Ply Width (in.)	8.0, 9.0, 9.5 depending on radial location
Axial Ply Length (in.)	2.00, 2.01-2.49, 5.00 depending on radial location
Overlap, Min. (in.)	1,5
No. Plies Pre-punched	124

Ply pre-punching (to fit over cells) was carried out

a. Make a cell array pattern of the comb tooth joint area by taking a carbon paper rubbing. Glue the rubbing to an aluminum strip of the same width and length as the width and circumference of one layer of plies. Drill 0.1 dia holes at cell locations.

b. Place one circumferential layer of overlapped plies in the punching jig (a small wooden jig specially made for this purpose).

c. Clamp aluminum template over plies and drill to duplicate cell array pattern.

After pre-punching, each layer was fitted over the cells, with the holes matched to their corresponding cells. This was a lengthy task.

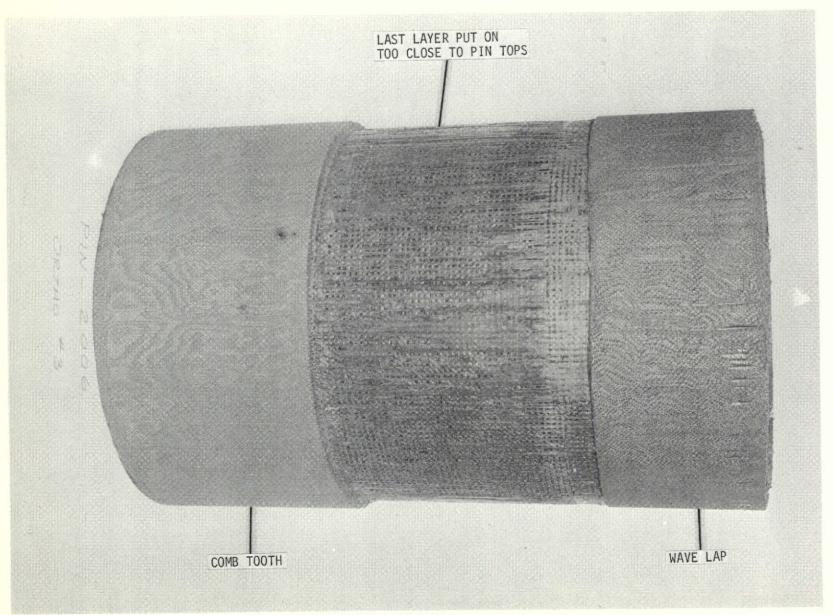


Figure 55. Ortho 3 - Cured State

During joint construction the lay-up was subjected to 8 autoclave debulks. Despite this, the wrinkle build-up between the cells in the comb tooth area was such as to preclude using all the cut plies. The tops of the cells were reached in 46 instead of 58 layers. The final OD was therefore reached prematurely.

Flexible cord was wrapped between the cells to prevent the unsupported lengths of the cells from being loaded by the bagging material during debulk and to transfer the load to the plies being debulked, instead. This wrapping was continued above the tops of the cells. The cord transmitted the debulking load past the cells to the lay-up.

After joint lay-up the joints were cured using the standard cycle in Table 7. Figure 55 shows Ortho 3 after cure.

3. Conclusions

The Interleave joint is not a practical idea and will be dropped.

Inside-out molding of orthogonal cylinders should be investigated to possibly improve quality and to save time (by allowing pressurization).

The performance of the Comb Tooth joint will have to be outstanding in order to out-weigh the many difficulties and the extra expense of its manufacture.

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APPENDIX A

SAMPLE CALCULATIONS

APPENDIX A

I. Calculation of Shape Change Factors f_{θ} and f_{z} .

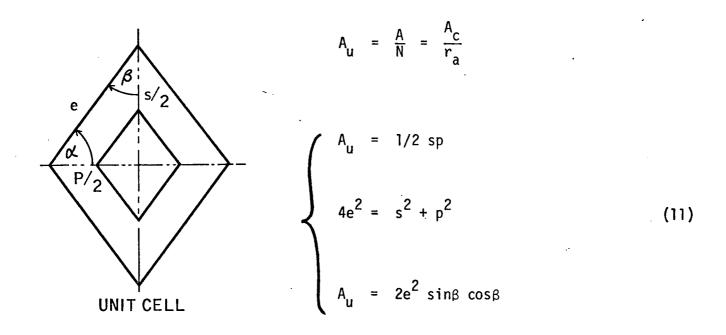
Refer to Figure 20. Neglect surface curvature. It is clear that the final circumferential and axial distances between cell centers are determined once r_a , the cell dimensions and the final cylinder dimensions are specified. Designate the circumferential spacing s and the axial spacing p. The number of cells is

$$N = r_a \frac{A}{A_c}$$
 (9)

where A = cylinder outer surface area and Ac = cell cross-sectional area. The area of a "unit cell" is

$$A_{u} = \frac{A}{N} = \frac{A_{c}}{r_{a}} \tag{10}$$

This unit cell is in the shape of a rhombus, with sides e, vertex angles 2β and $180^{\circ}-2\beta$, minor diagonal p, and major diagonal s.



From Reference (15),

$$\sin\beta \cos\beta = \frac{1}{2}\sin(\beta + \beta) + \frac{1}{2}\sin(\beta - \beta)$$

$$=\frac{1}{2}\sin 2\beta$$

$$A_{u} = e^{2} \sin 2\beta \tag{12}$$

$$e^2 = \frac{A_u}{\sin 2\beta}$$

Therefore,

$$\begin{cases} s^2 + p^2 = \frac{4A_u}{\sin 2\beta} \\ s = \frac{2A_u}{p} \end{cases}$$
 (13)

Solving for p yields

$$p^4 - p^2 \left(\frac{4A_u}{\sin 2\beta}\right) + 4A_u^2 = 0 \tag{14}$$

Let

$$x = p^2$$
, $a = 1$, $b = -\frac{4A_u}{\sin 2\beta}$, $c = 4A_u^2$

Using the quadratic formula,

$$x = \frac{-b \pm \sqrt{b^2 + 4ac}}{2a}$$

Yields

$$p^2 = 2A_u \left[\frac{1}{\sin 2\beta} + \sqrt{\left(\frac{1}{\sin 2\beta}\right)^2 - 1} \right]$$
 (15)

The correct root must now be selected.

Since β has the range,

$$0 < \beta < \pi/4$$

investigate the roots over this range only. To be physically meaningful the roots must be real and positive. That is, for any β in the range of interest,

$$\left(\frac{1}{\sin 2\beta}\right)^2 - 1 > 0 \tag{16}$$

$$\frac{1}{\sin 2\beta} \pm \sqrt{\left(\frac{1}{\sin 2\beta}\right)^2 - 1} > 0 \tag{17}$$

It can be easily shown that for the range of β selected (16) is always ≥ 0 . However, it can also be shown that, regardless of the choice of the sign of the radical in (17), (17) is ≥ 0 . This sign must therefore be selected on other grounds. By inspection for $\beta=30^{\circ}$,

$$s = p = e \tag{18}$$

and therefore,

$$Au = \frac{\sqrt{3}}{2} e^2 \tag{19}$$

Substituting

$$p = \sqrt{2(\frac{\sqrt{3} e^{2}}{2}) \left[\frac{2}{\sqrt{3}} + \sqrt{(\frac{2}{\sqrt{3}})^{2} - 1}\right]}$$

$$p = e \sqrt{\sqrt{3} \left[\frac{2\sqrt{3}}{3} + \frac{\sqrt{3}}{3}\right]}$$

$$p = e \sqrt{2 + 1}$$

Therefore the minus sign must be selected:

$$p = \sqrt{2A_u \left[\frac{1}{\sin 2\beta} - \sqrt{\left(\frac{1}{\sin 2\beta}\right)^2 - 1\right]}}$$
 (20)

$$s = \frac{2 A_u}{p} \tag{21}$$

$$A_{u} = \frac{A_{c}}{r_{a}}$$
 (10)

Equations (10), (20) and (21) define the axial pitch, circumferential spacing, and unit cell area for any selected β , A_c and r_a .

To calculate the shape change factors f_z and f_θ deform the rhomlus into a square such that the vertices at the ends of the minor axis (p) can move only along that axis and the vertices at the ends of the major axis (s) can move only along that axis. The length of the side e will be unchanged. The helix angle for the square is β_1 = 45° and for the original rhomlus is β_2 = 30°. For these angles we have

$$A_{u} = \frac{\sqrt{3}}{2} e^{2}$$

$$Au_0 = e^{2}$$

From the check calculation

and therefore

$$s = \frac{2(\frac{\sqrt{3}}{2}) e^2}{e}$$

$$s = \sqrt{3} e$$

For β_1 = 45°, substituting in (20) yields

$$p_0 = e \sqrt{2[1 - 0]}$$

$$p_0 = \sqrt{2} e = s_0 \text{ (square)}$$

Finally, from (1) and (2), recognizing that p and s are proportional to 1 and d, respectively,

$$f_3 = \frac{p}{p_0} = \frac{e}{\sqrt{2} e}$$

$$f_z = \frac{0.707}{}$$

$$f_{\theta} = \frac{s}{s_0} = \frac{\sqrt{3} e}{\sqrt{2} e}$$

$$f_A = \frac{1.225}{}$$

The actual values of \mathbf{p}_0 and \mathbf{s}_0 used will vary slightly from those just computed. This is because the number of indexing gear teeth and the number of lay-up machine lead screw threads/inch selected must be integers.

II. Calculation of Theoretical (voidless) Cell Density

Let W_c = cell weight, W_r = resin weight and W_f = fiber weight, in a cell.

$$W_{c} = W_{r} + W_{f}$$
 (22)

Since

W = ρ V, where ρ is density and V is volume,

$$\begin{cases}
\rho_{c} V_{c} = \rho_{r} V_{r} + \rho_{f} V_{f} \\
V_{c} = V_{r} + V_{f}
\end{cases}$$
(23)

These equations yield,

$$\rho_{c} = \rho_{r} \frac{V_{r}}{V_{c}} + \rho_{f} \left(1 - \frac{V_{r}}{V_{c}}\right)$$

$$= \rho_{f} - \frac{V_{r}}{V_{c}} \left(\rho_{f} - \rho_{r}\right)$$
(24)

The resin weight fraction in the cell is

$$R_{W} = \frac{W_{r}}{W_{c}} = \frac{V_{r} \rho_{r}}{V_{c} \rho_{c}}$$
 (25)

Substituting in the previous expression for $\boldsymbol{\rho}_{\boldsymbol{C}}$

$$\rho_{c} = \rho_{f} - R_{W} \frac{\rho_{c}}{\rho_{r}} (\rho_{f} - \rho_{r})$$
 (26)

Collecting terms and solving for $\rho_{\boldsymbol{C}}$

$$\rho_{C} = \frac{\rho_{f}}{1 + R_{W} \left(\frac{\rho_{f}}{\rho_{r}} - 1\right)}$$
 (27)

Calculation of Theoretical ρ_c

Yarn received under PO NO1417 $(\overline{\rho}_f = 1.327 \frac{gm}{cc})$

$$\rho_{c} = \frac{1.327 \text{ gm/cc}}{1 + .32 \left(\frac{1.327}{1.30} \text{ gm/cc} - 1\right)}$$

$$\rho_{\rm c} = 1.318 \frac{\rm gm}{\rm cc}$$

For PO NO1336 ($\rho_f = 1.283$)

$$\rho_{\rm c} = 1.288 \frac{\rm gm}{\rm cc}$$

III. Resin Content Estimate of % Collimation Rejection (Assume Normal Data)

Basis for rejection: Specification AGC-902292, Fabric Graphitized, Resin Impregnated, paragraph 3.2.1.3. Paragraph requires resin content \pm 2% in pre-preg fabrics. Collimated strands are a form of pre-preg, although not a woven one.

Collimation Data (GE Program STATAN***)

$$N = 57$$

 \overline{X} = 53,77% Resin

$$S = 5.85\%$$

Calculation:

$$t = \frac{X - \overline{X}}{S} = \frac{55.77 - 53.77}{5.85} = \frac{2}{5.85}$$
 (28)

From Normal Distribution Table (Reference (15))

$$F(.3584) = \int_{-\infty}^{-\infty} f(t) dt = 0.6400$$
 (29)

$$F(o) = \int_{-\infty}^{0} f(t) dt = 0.5000$$
 (30)

$$F(.3584) - F(0) = 0.14$$
 (31)

Likewise

$$F(o) - F(-.3584) = 0.14$$

Therefore

$$[F(.3584) - F(o)] + [F(o) - F(-.3584)] = 0.28$$

and

Coeff of variation
$$\frac{S}{X}$$
 = .108 \approx 11%

IV. <u>Volatile Content Calculation % Collimation Rejection</u> (Estimate) (Assume Normal Data)

Basis for Rejection: AGC-902292, Paragraph 3,2,1,3 requires (by implication) volatiles controlled to \pm 1.5%.

Collimation Data (GE Program STATAN***)

$$N = 59$$

$$\overline{X} = 7.28$$

$$S = 1.33$$

Calculation:

$$t = \frac{X - \overline{X}}{S} = \frac{8.78 - 7.28}{1.33} = \frac{1.5}{1.33}$$

$$t = 1.128$$

% Rejection ≡ R

$$100 - R = 200(F(1.128) - F(0))$$

$$R = 100 - 200 (.8700 - .5000)$$

R = 26% Coef. of Variation
$$\frac{S}{X}$$
 = .183 \approx 18%

V. Estimate of Resin Content

After Filament Winding (wts in gms)

Helix 3

Yarn Spool Weight Change (ΔW_S)	1340.6
Waste Yarn (W _W)	-136.0
Filament Weight (W _f)	1204.6
Wound Cylinder (W _c)	5690,0
Backed Array (W _a)	-2812.0
Total Weight Gain (ΔW _t)	2878.0
Filament Weight	-1204.6

Resin Weight After Winding (W_r) 1673.4

2005 Wte before debulk

$$W_{BD} = W_{LD} - \overline{\Delta W}_{LD} = 3980 - 146 = 3834 \text{ gm}$$
 (32)

Wte of cells

$$W_{RC} = \rho_{RC} V_{RC}$$

$$= (1.29 \frac{gm}{cc}) V_{p}$$

$$V_{C} = (.0016 in.^{2}) (.75 in.) (2.54)^{3} \frac{cm^{3}}{in.^{3}}$$

$$= 0.0197 cm^{3}$$
(33)

$$W_C = (0.0197 \text{ cm}^3) (1.29 \frac{\text{gm}}{\text{cm}^3})$$

$$W_C = 0.0254 \text{ gm}$$

$$W_{C_T} = N_C W_C$$

$$N_{C} = \frac{.18 \text{ A}}{A_{R_{C}}} = \frac{(.18) (17.02 \text{ in.}) (7.5 \text{ in.}) (3.14)}{.0016 \text{ in.}^{2}}$$
(34)

$$N_{c} = 45093$$

$$W_C = (45093) (.0254 \text{ gm}) = 1145 \text{ gm} (\frac{.625}{.75})$$

$$W_{C_T} = 954 \text{ gm}$$

Weight of backing

$$W_B = W_A - W_{C_T} = 2812 - 954$$

$$W_B = 1858 \text{ gm}$$
(35)

Weight of Wound Array less Backing

$$W_{C}^{\prime} = W_{C} - W_{B} = 5690 - 1858$$
 (36)
 $W_{C}^{\prime} = 3832 \text{ gm}$

Resin Content After Filament Winding

$$R_{F} = \frac{W_{R}}{W_{C}} \cdot 10^{2} = \frac{(100) (1673.4) \text{ gm}}{3832 \text{ gm}}$$
 (37)

$$R_{\mathsf{F}} = 43.7\%$$

Resin Pick-up During Impregnation

$$W_{R_{I}} = W_{BD} - W_{C}^{-} = 3834 - 3832$$
 (38)
 $W_{R_{I}} = 2 \text{ gm}$

% Resin After Debulk

$$R_{D} = \frac{W_{R} + W_{R_{I}} - \overline{\Delta W_{LD}}}{W_{LD}} = 10^{2}$$

Where ΔW_{LD} is the average weight loss of Helix 1 and Helix 2 in lateral debulk. W_{LD} is the cylinder weight after lateral debulk.

$$R_{D} = 100 \left(\frac{1673.4 - 146}{3980} \right)$$

$$R_{D} = 38.3\%$$

2. Following similar procedures for the remaining cylinders yields the results in Table 27.

TABLE 27
ESTIMATED CYLINDER RESIN CONTENTS

	After Filament Winding	After Lateral Debulk	
Cylinder	R _F (%)	R _D (%)	Comment
2001	-	42.0	Poor est.
2003	-	45.0	Poor est.
2005	43,7	38,3	Good est.
2002	46.7	NA	Poor est.
2004	46.8	· NA	Poor est.
2006	38,4	NA NA	Good est,

3. For all cylinders

$$\overline{R}_{F} = \frac{175.6}{4} = 43.9\%$$

4. For Helical cylinders

$$\overline{R}_p = \frac{125.3}{3} = 41.7\%$$

VI. Joint Length Reservation

Assume full cylinder is pulled axially. In the AGCarb half of joint just before failure:

$$P_z = 2 \pi r t F_{tu}$$
 (40)
 $P_z = 2 (3.1416) (4.25 in.) (0.5 in.) (5,000 $\frac{1bt}{in.2}$)$

$$P_{7} = 66,000 \text{ lbf}$$

$$\sigma_{S} = \frac{P}{2\pi \overline{r} 1} \leq F_{SU} = 1000 \frac{1bf}{in.^2}$$
 (41)

$$1_{\text{required}} \ge \frac{P}{2\pi \, r \, F_{\text{su}}} = \frac{66,000 \, \text{lbf/in.}^2}{2(3.1416) \, (4.25) \, (1000) \, \frac{\text{lbf}}{\text{in.}}} = 2.5 \, \text{in.} (42)$$

VII. Type III Joint Shear Area Increase Derivation

Let

m = No. bond surfaces

n = No, of fil, layer

m = 2(n-1)

t = thickness of each layer

Diameter of each layer =
$$d_1$$
 + 2 i t i = 1.0 \rightarrow m (43)

$$A = \pi \int_{i=1}^{m} (d_{i} + 2 i t)$$
 (44)

=
$$\pi 1[d_1 m + 2 t \frac{m}{2} (m + 1)]$$

=
$$\pi l m [d_1 + t(m + 1)]$$

=
$$2\pi 1(n - 1) [d_1 + (2n - 1)t]$$

$$\frac{A}{A_0} = \frac{2(n-1) [d_1 + (2n-1)t]}{(d_1 + .5)}$$
(45)

$$= 2(n-1) \left(\frac{\frac{1+(2n-1)t}{d_1}}{1+.5/d_1} \right)$$
 (46)

VIII. Sizing of Interlock Joint

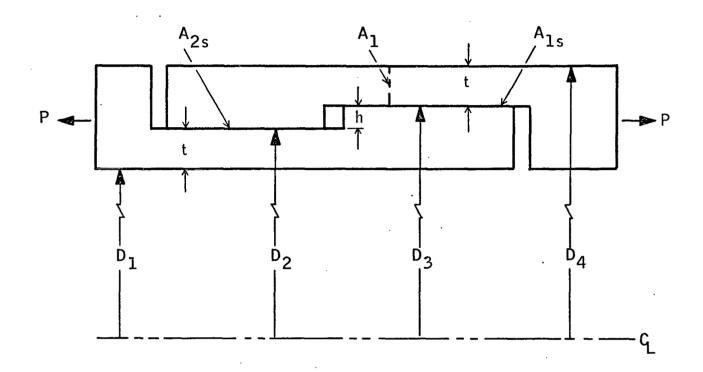


Figure 56
Idealized Interlock Joint

Assume:

$$A_{1s} \equiv A_{2s}$$
, $t_1 = t_2 = t$, neglect bending

Criteria:

$$2A_{2s} F_{sU} \geq A_1 F_{tu}$$

Assume (Table 11)

$$\frac{2A_{2s}}{A_{1s}} \geq \frac{F_{tU}}{F_{su}} = 5 \tag{47}$$

$$A_{2s} \geq 2.5A_{1}$$

$$A_{2s} = \pi D_{2} 1_{2s}$$

$$A_{1} = \frac{\pi}{4} (D_{4}^{2} - D_{3}^{2})$$

$$\pi D_{2} 1_{2s} = 2.5 \frac{\pi}{4} (D_{4}^{2} - D_{3}^{2})$$

$$1_{2s} = \frac{5}{8} (\frac{D_{4}^{2} - D_{3}^{2}}{D_{2}})$$

$$(48)$$

Calculation, for:

$$D_{1} = 8 \text{ in.; } t = .1, .2, .3; h = .1$$

$$\frac{t = .1}{1_{2s}} = 5(.1) \left(\frac{8 + .4 + .2}{8 + .2}\right) = .5 \left(\frac{8.6}{8.2}\right)$$

$$\frac{t = .2}{1_{2s}} = 5(.2) \left(\frac{8 + .8 + .1}{8 + .8}\right) = \left(\frac{8.9}{8.8}\right)$$

$$\frac{t = .3}{l_{2s}} = 5(.3) \left(\frac{8 + 1.2 + .2}{8 + 1.2}\right) = 1.5 \left(\frac{9.4}{9.2}\right)$$

1_{2s} = 1.01 in.

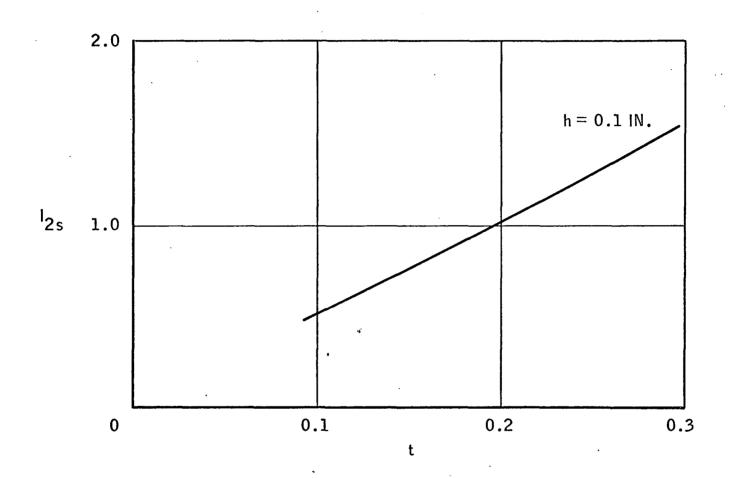


Figure 57 Shear Length vs. Thickness - Interlock Joint

APPENDIX B

HISTORICAL INSPECTION REPORTS

AFFENDIX BI INSPECTION REPORT

Nº 512310

165 SKETCH ATTACHED FI 45CS 0721-3 (REV 1/71) I NEW S/H /// G 5/2 & FACILITY AND INSP. AREA 4. PART/HATERIAL HARE YNVACAIGLD 2. HO. BAY YTT. | 3. PART HO. 5 | 19 | 7 | 1139 509 IT CYLINDER (HELICAL) 2001 D1 150 2019 143 HODEL 8. INSPECTION CRITERIA OTHER (EXPLAIN) 6. Q CAL 7. W.O. NO. <u> 1190-44</u>-302 DWG. SPEC. LL. 12. 077. NOT ACC. 15. CONTRACT NO. S. CHIT OF ETAS. | 10. LOT SIZE 14. 5.0. 19. NA II. GTT. HISP. 96-1-6-1 NA 15. BUT CO. 16. SUPPLIER HAVE AND CODE 17. DISTR. | 16. P.O./TWA NO. IDO 961161 12. GFP . [] 84. BEXT ASSY P/H 31, FREVIOUS REPORT KO. 22. YEST NUMBER FR. PRE TEST сен 🛛 TEST MAc 2001 est 📋 10. DISCREPANCY OR NON-COMPORNANCE CL OF CHAR, zi. Pastin. LIST IN ORDER: NO. PCS. - DWG/SPEC REQ - INSP. RES. - ITEM IDENT. becision REDESIGNATE 1/39309-1 1139309 REQUIRES -3 Assy AS 1139309 MODZ (SHORTE TO BE 17.02 IN LONG. PCB) AND 1139309 MOD 10 LAID ()P IN TWO PCS ABOUT (LONGER PC). PROCESS AS 3IN AND 14 IN LONG, RSP. MALFUNCTION OF LAY-UP MACH-SEP CYL. CHANGE INE CAUSED ONE CIRCULAR POW MFG SIN (SAME AS PROCESS OF CELLS TO BE OUT OF Pas-I.D. # 1 TO 2001 MOD 2 TION SPEC By 1139309 (SAT AND 2001 MODIO. ESTABLISH SEPARATE PROCESS HISTORY 4). REQUIRED SEFARATE -3 REDUCES FOR EA CYL. PERCO AT BOD ROW OF CELLS. CONFORM TO MEMORIMISUM DTD 3 May N B500: M1263 1971. PROC./HFG. KEPR. 12. PHILATOR IDENTIFIE DATE HACHER DATE 25. ER/HR INSPECTION-ISENTITY OTT. ACC. QTY. NOT ACC. FACILITY AND INSP. AREA NEW I.R. NO. DATE 24. REIN-OTY. OTHER (EXP.) | EXPLANATION OF OTHER OTY, NOT ACC. DATE QTY. ACC. 25. EP/NR BESUBHITTAL TECISION ENG. REPR. DATE cust. DATE 26. CORRECTIVE ACTION ACTION-TO PREVENT RECURRENCE 1. IROUSUE SACOT WILF OF LAY-UP MACHINE MACHINE 2. GO TO HAND LAY-UP IN INTERIM RESPONSIBILITY QTY. RIS. OTY. SALVACE PROC./HEG. RLPH. OC BEPR. DATE TATECTED ANTERIAL ACTION Acc . SUPPLIER

COMPARY

ISS INSPECTION REPORT

Nº 512438

SKETCH ATTACHED [] 1205 0721-3 INCV 1/711 FOR RECORD DULY - WRITTEN TO DICUMENT EXPERIMENTAL OFFICIALS PAGE 1 OF L FACILITY AND INSP. AREA 4. PART/HATEPIAL HADE INTRENELD JIL I ITEN S/H MrG SIN Z. NO. DAY YR. S. FART HO. DASI: C/L 2019 12 27 71 1139 309 CHELICAL CYLINDER. 2001 MEDEL 8. 193FECTION CRITERIA M enc. SPEC. DIA. NO. . THIT OF HEAD. | 10. LOT SIZE 14. 5,0. #3 NA SNP-1 NA NA NΑ 12. BUT CO. 16. SUPPLIER NAME AND CODE 17. DISTR. | 15. F.C./IVA #6. 27. ALRC IDO 961161 Zo. GFP [] SI. PREVIOUS FEPGRY CO. S C. HEXT ASST BIN SE. KEKT ASSY S/A 32 TEST BUFBER 1.3 PLE FEST 12. ms sin Gru D 7257 NA NA 2001 csi [] to. Discrepancy or non-conformance 20. DISPOSITION/COMMENTS CHARL CL. THE OF 30. CHARL REVIEW DECISIONS DECISION REMOVE COMB-TOOTH JOINT 1139309 Reguless -2 Assy to HAVE UNDAMHGED "COME TOOTH" JOINT AREA WITH "STRAINIT LAP" JOINT, MAIG NO AND FLAMENT WINDLERS, "BALDONING" OF MORE COME TOOTH JOINTS ON HELICOL CYLE INTERNAL PRESENCE BAS DURING LATERAL DE PRESENT MOLD RULEAGE WITH BULK SEVERFLY DOSCREPLYL, WALL & MIC-ALIGNED CHIS IN THIS ASSO. RESIN TO TRAP INFLEW & Poepline COLOGO DEEP RESIN PUDDLES HERE ALGO Rose Mars Pearles RULED UP DUTTE FLAM-PAT WINDING LAYER CYLINDERS 1139 309 RECOURES . 2 to HAVE CYLINDRICAL DNGER IN LATERNA FIXTURE BHAPE. POST-LATERAL LINE HOURGIASS PROC./HTG. REPR. FATE 1125/12 DATE 23. ER/ER APPROVALS FACILITY AND INSP. AREA QTY. ACC. GIT. HOT ACC. HEW I.R. KO. INSPECTOR IDENTITY DATE 14. REIN-SPECTION QTY. ACC. CTY. NOT ACC REHTO THER (EXP.) | EXPLANATION OF OTHER QC PEPS. EATE ZE FE/NR ZESUENITTAL ENG. REPR. DATE 350151011 26. CORRECTIVE ACTION ACTION TO PREVENT RECURRENCE CA. SE CE BISCREPLROY 1. NEWFFICIENT COMB TOOTH HEER WALL THICKNES, 12/27/71 FOCE MULL RELEASE AGENT, & AND "WELL" EFFECT 1,2,3,4.: JOINT ARIST. SEE "REVIEW DECISIONS" ABOVE. INSUFFICIALT BYILDING OF CYLINDER DURING 4. PROPER EXHIVE FOURE PRESSURE PROC./HES. REPR. OC REPR. DATE REJECTED SUPPLIER

INSPECTION REPORT - Cont. Sheet

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3	1	1139309-Z REQUIRES RADIAL	3	ATTEMPT SUPERSTAGE "CYLL TO STR-
	1_C	CELLS POINTING ALONG PADIT & IMPLIES		ENGTHEN ROOM APPLYING 200 PEIG. IN
	<u> </u>	UN-WRINKLED FILAMENT WINDINGS.		VESTILATE MORNIE TOUT AFTER CUEE O
	<u>[</u>	JUINT PLY DEBULKING AND CYLINDER		DEEDLEWIG AT LESS THAN 200 pers.
	1 1	ASSEMBLY CLUE OUSES EXTENSIVE	<u> </u>	("BUFFTS TAILING" NA TO NEUX- > DUE
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	k	CLING OF WINDINGS.	<u> </u>	
			 	
1	1	113930-2 REQUIRES OD = 9.07 IN AUD	A	MOOKET AS IS. RELIVERS DIMENSION
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and in the little to the INSPECTION REPORT TING COMPANY Nº 512438 SKETCH ATTACHED [] 1. NO. PAY YR. 3. FART HO. 4. PART/HATERIAL HADE INTRECIOLD THE TEN S/H MFG 5/W 2019 12 27 71 1139564 CYLINDER (HELICAL) . UUZL SPEC. | 1.1. 143 D 0113. \$. .AIT OF BEAS. | 10. LOT SIZE 12. CTY. HOT ACC. 13. CONTRACT NO. 51, Cri. 185P. 14. 3.0. NO. NA SNP-1 NA MΑ NA 15. 127 CO. | 16. SUPPLIER LANE / 12 CODE 17, GISTE. | 10, P.O./108. ED. IDO 961161 ALRC DE, HEXY ASTEY SAN MFG 34. NEXT ASSY P/H 25. GF# [] 21. PRIMING REPORT NO. 32. YEST MUUUER PRE TUST SPH D NA NA 5/4 2003 zo, bisposition/coruents AS. BILGREFANCY OR NON-CONFORMANCE r CL G OF GIMB. [] cr 1134564-1 REQUIRES CONTINUOUS Accert As 1s. TAKE JOINT AXIAL CYLINDOICAL SURFACE: PART HAS SPECIMENS FROM CUMPAMAGED DEEP CLOSUMERENTIAL GROOVE IN IN-TREADID IN THREAD LOCK " JOINT AREA 113956 9-1 REGUIRES PADING CALLS PO-SUPERSTACING NOT DONE DUE DESIGN (INTING ALONG RADIT & IMPLIES UN-"LEDIF LAP" JOINT. ACCEPTOR AS IS, COTTONE FREGRACI AS FOR LYTIN 3 ON WEINHERD FLAMENT WINDLES. EXTENSIVE PODIAL COLL TILT DISSETURD. THITED IR 512938A CELLS STORE WINDING DETORTION IN TILT DIRECTION, 1139564-1 ALSTMELY USES WITE-ACCEPT AS IS. WILL NOT AFFERT INTEN MOLD CYL 118958-1. LATTER DED DEE PROC./BFG. REPR. 1/25/72 DATE SS. ER/HD APPEOYALS INSPECTOR IDENTITY OTY. ACC. OTY. HOT ACC. KEW LE. NO. FACILITY AND IMPP. AREA DATE 12 1517-STECTION ' QTY. OTHER (EXC.) | EXPLANATION OF OTHER DIT. ACC. QTY. HOT ACE. QC CEPR. DATE e el/er Decheritial Besieve FAC. ETPR. DATE CUST. ZG. CURPECTIVE ACTION ACTION TO PREVENT RECURRENCE CALSE OF DISCREFAROY 1. MACHINING ERREVE I. CLEGGE GUPTEVISION). 12/27/71 2. 200 POIL CURE PRESSURE 2. SEE JR CIZ488A. MEM 3 3. SAMEAS 2. 3. SAME AS 2. RESPONSIBILITY OTY. RTS. FROC./MFS. REPH. DATE QC AEPR. DATE ESTERTICE ESPECIATION SUPPLIER

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INSPECTION REPORT - Cont. Sheet 169

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HISTORICAL OPTICATION I

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Nº 512438

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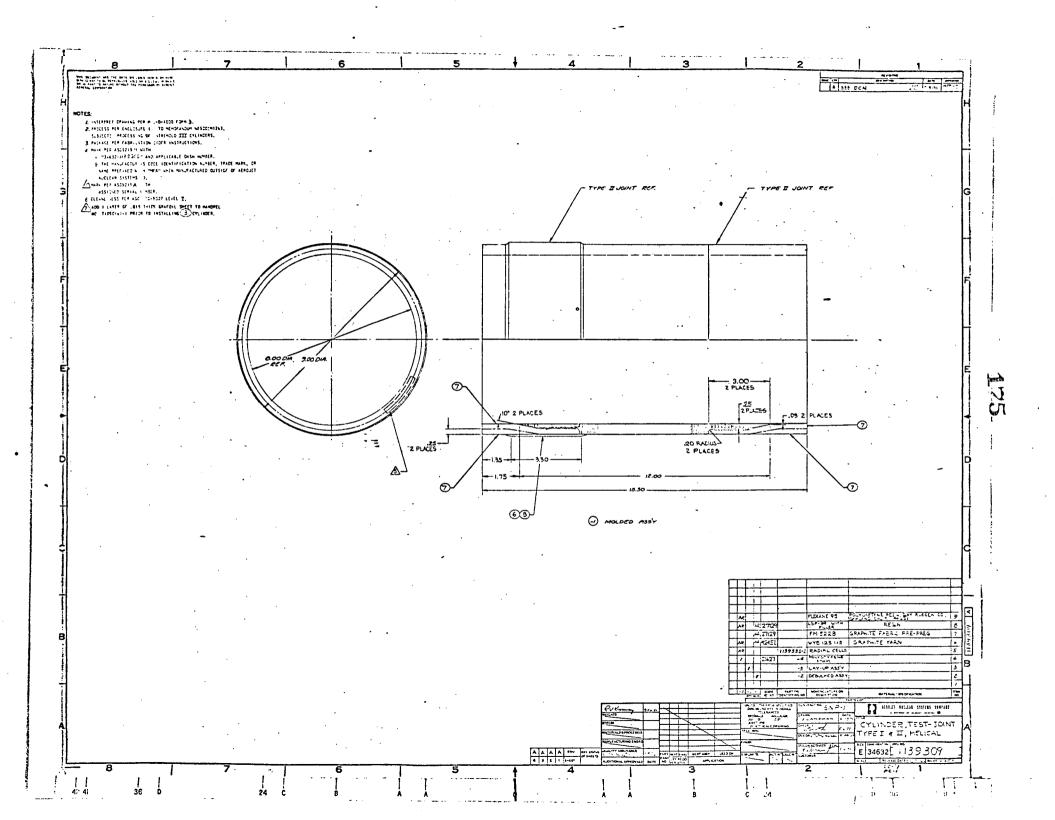
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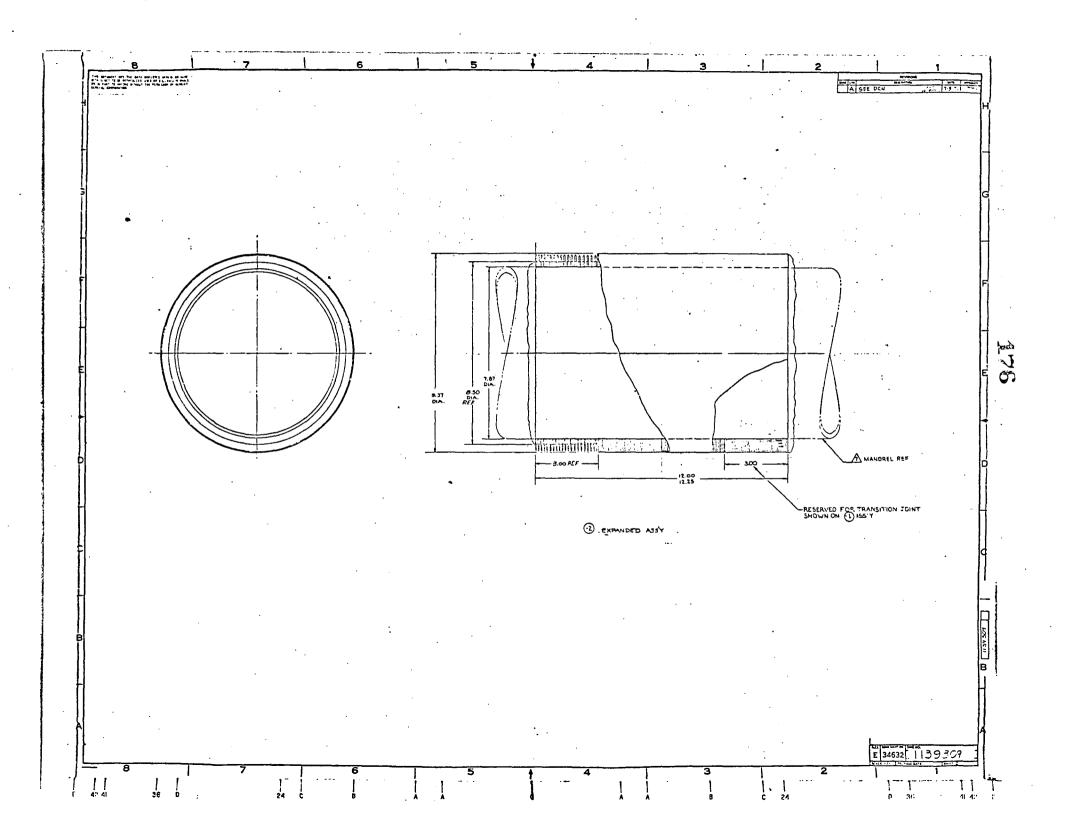
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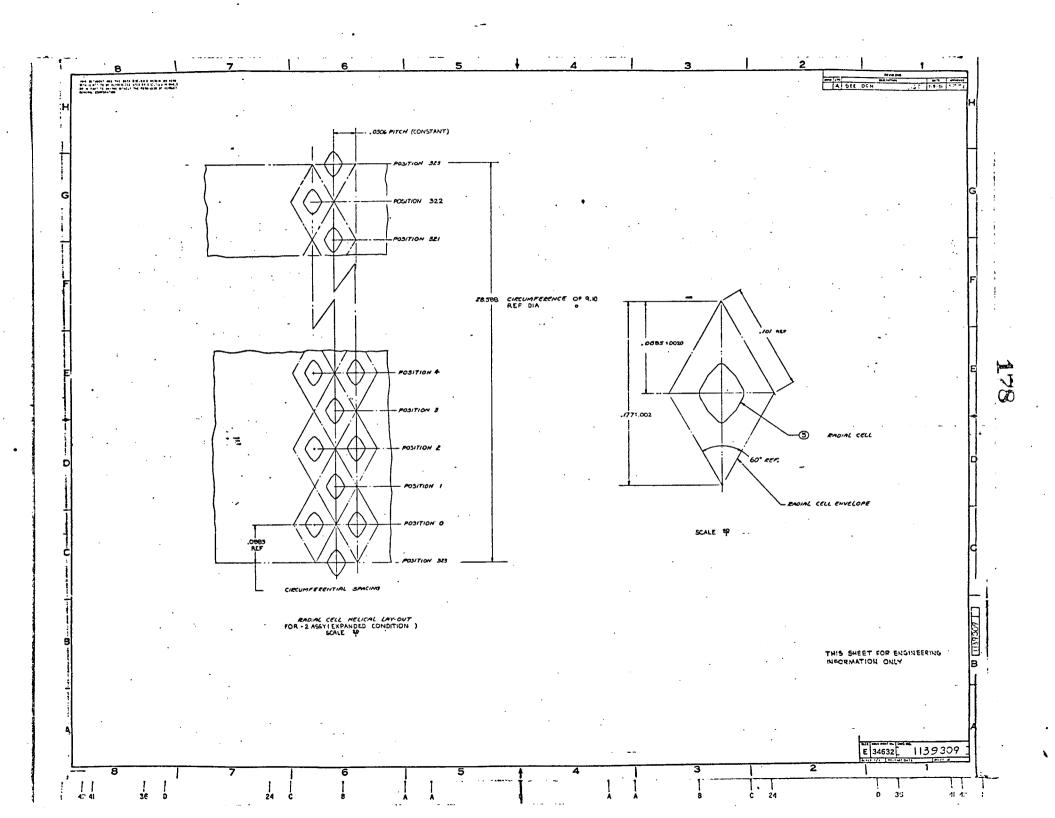
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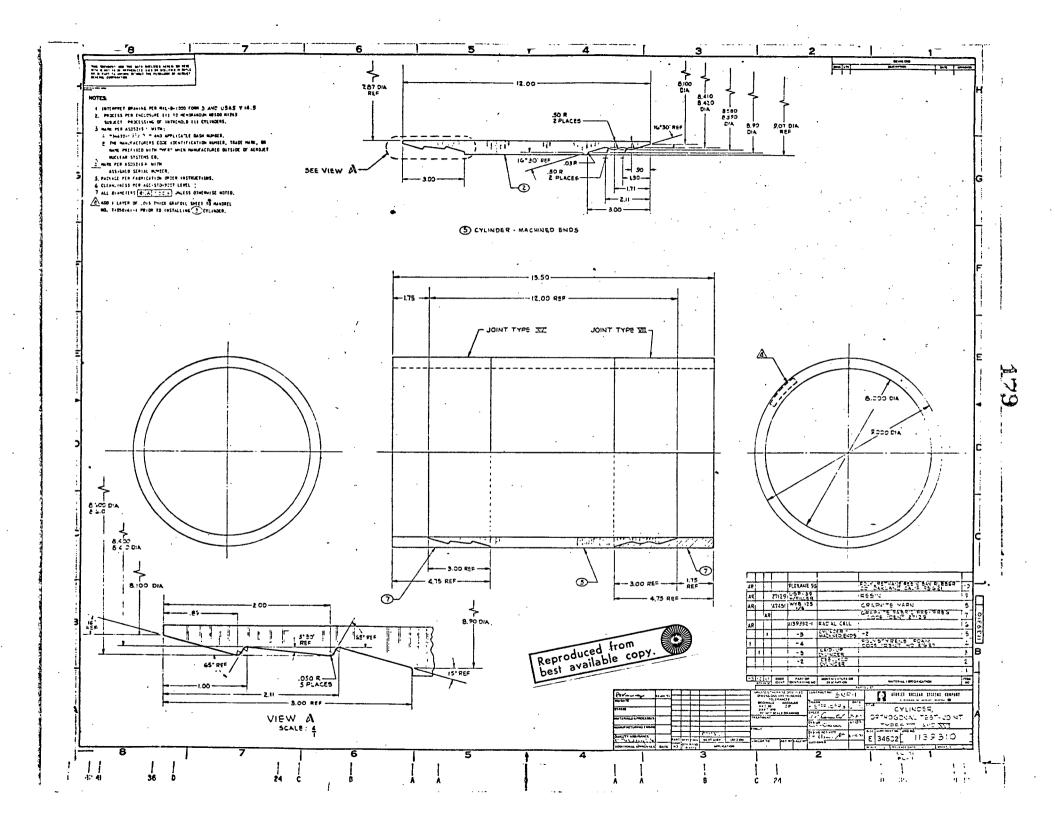
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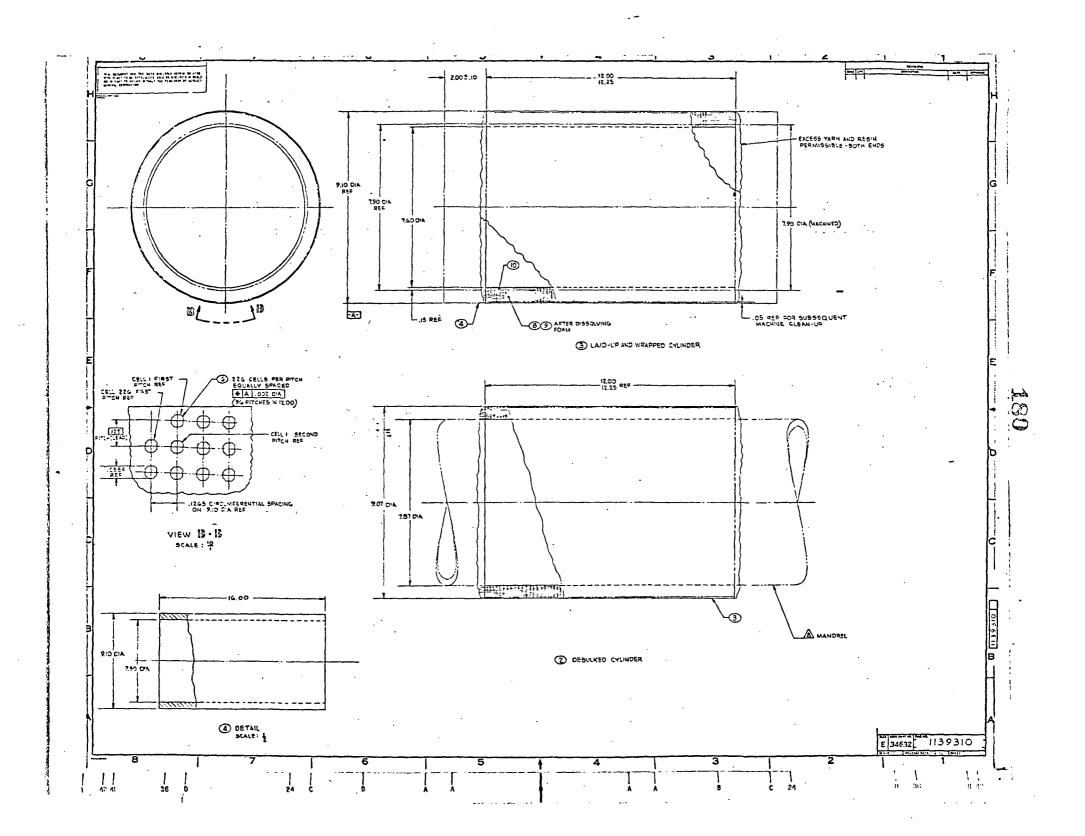
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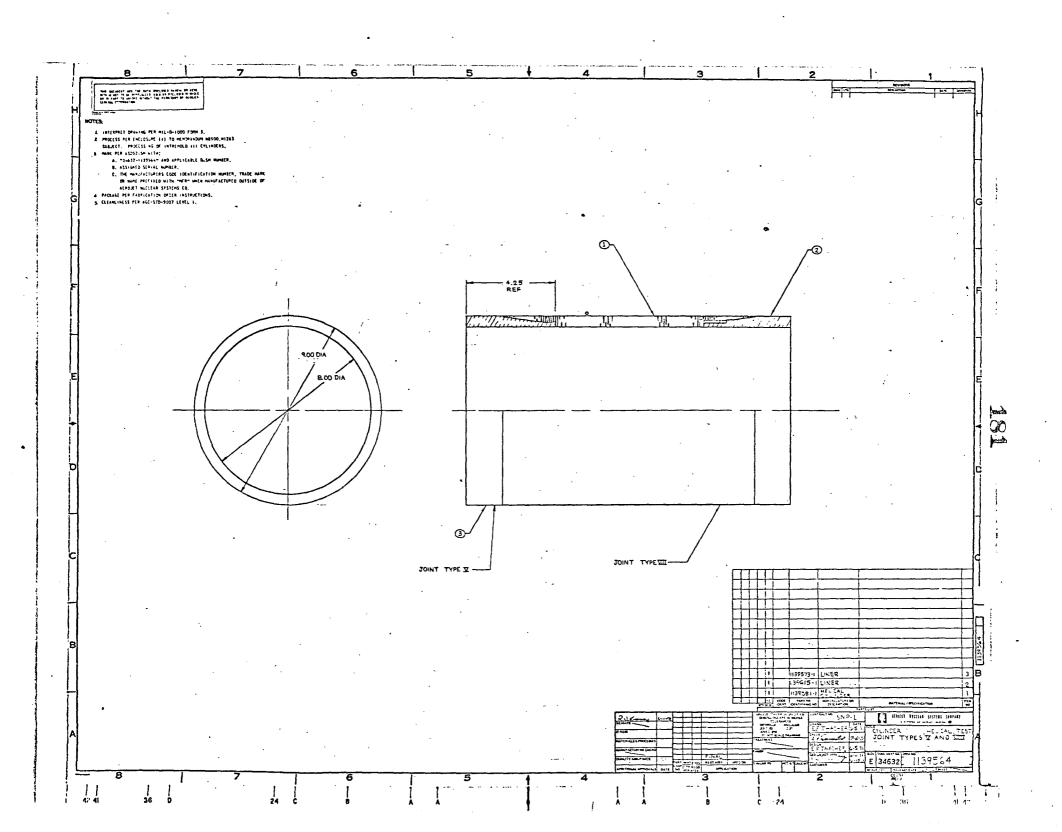


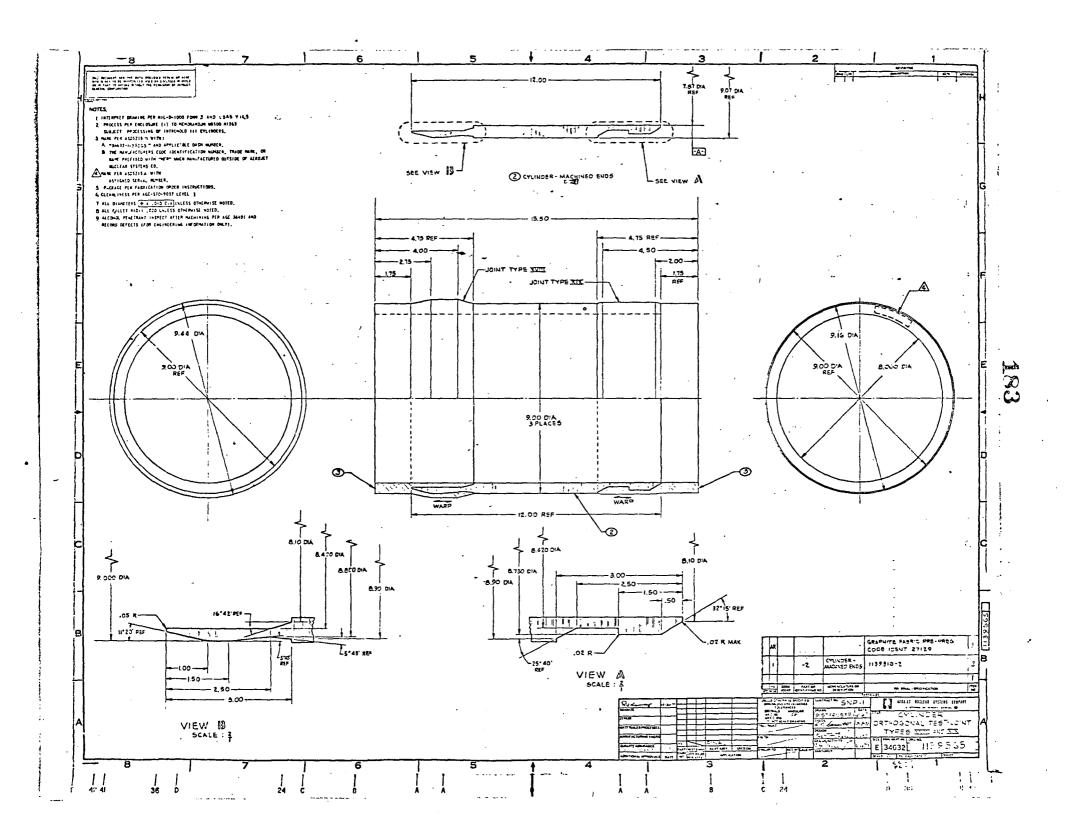


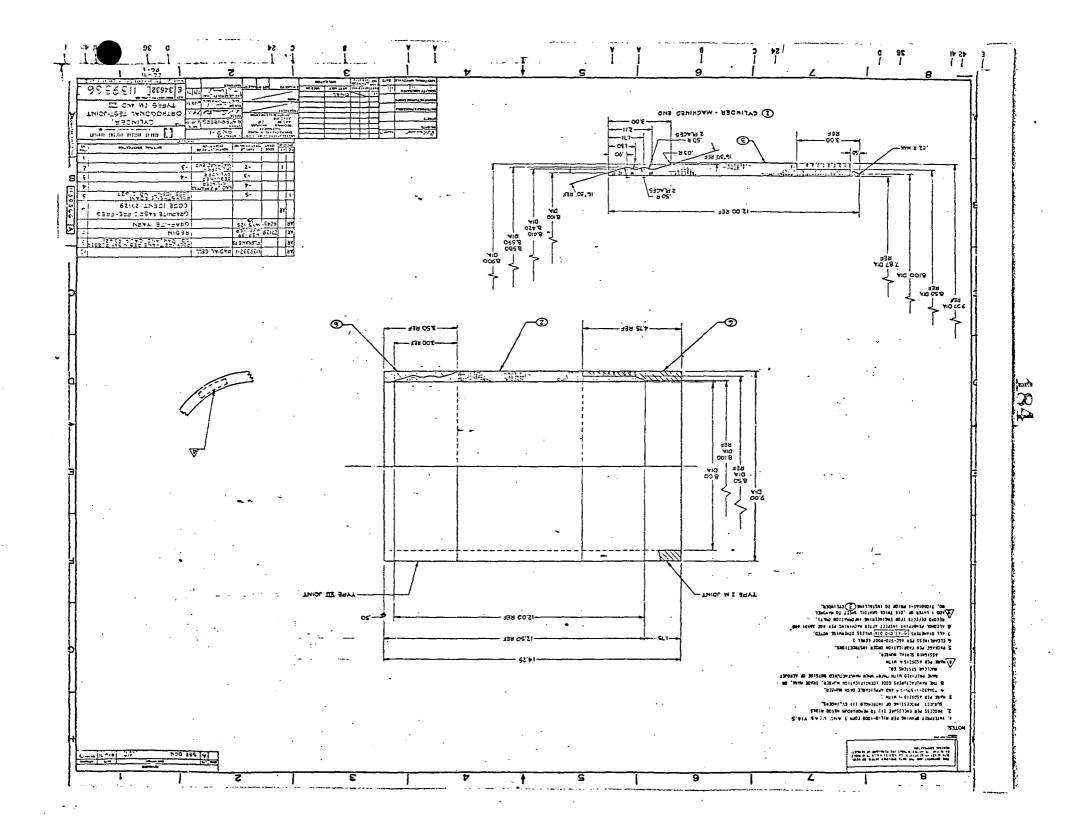


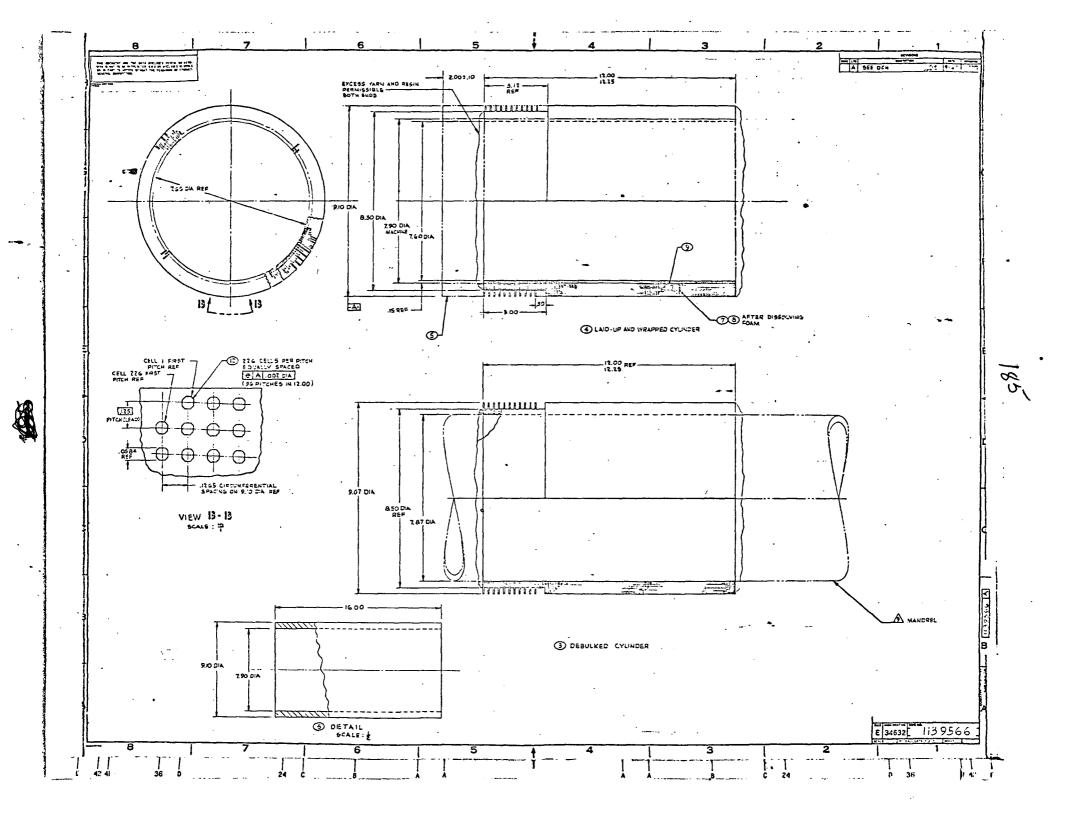


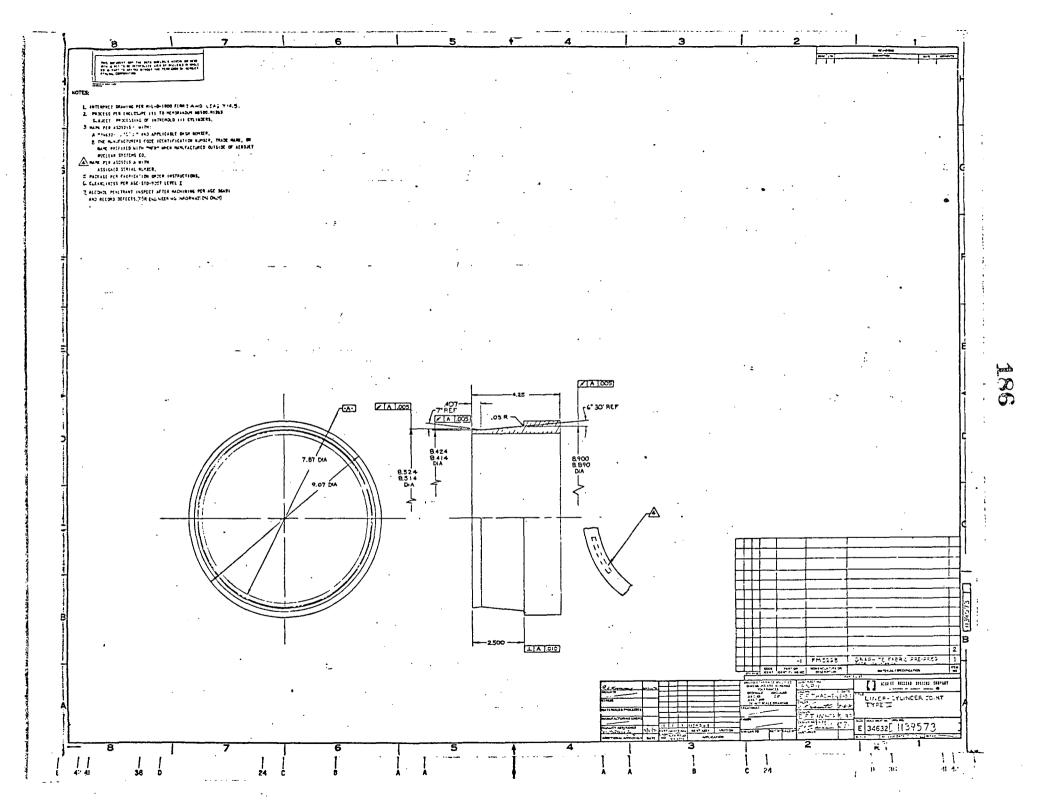


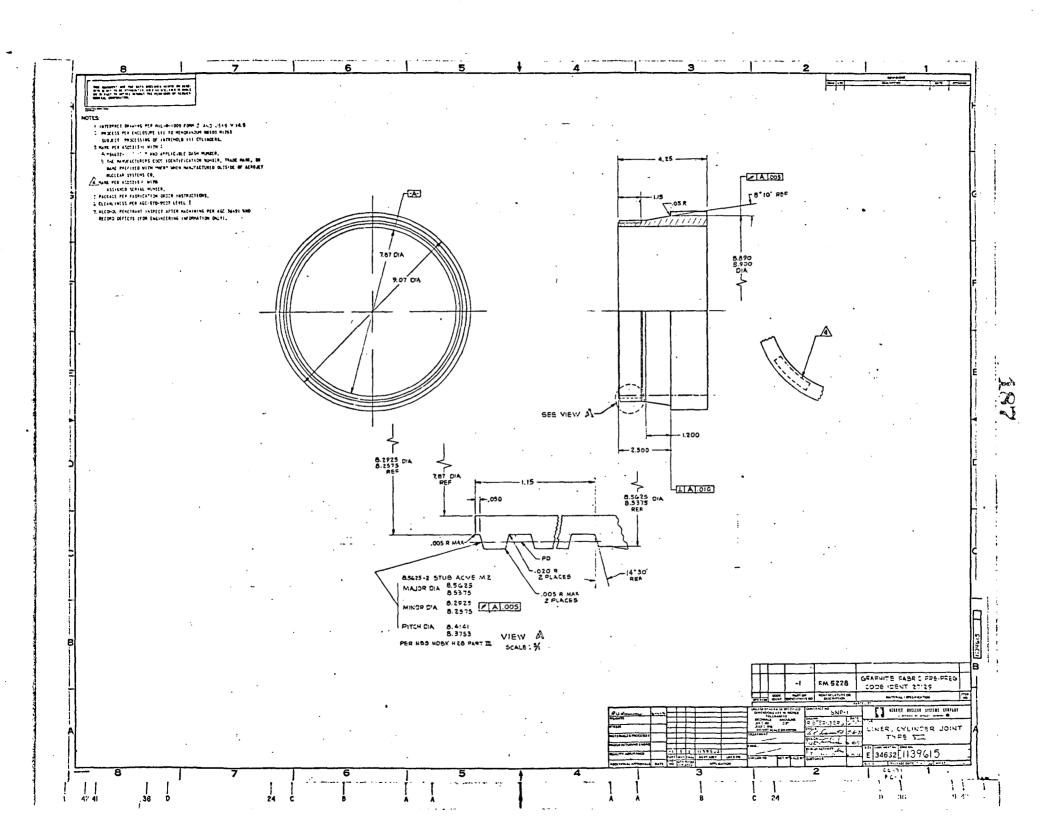


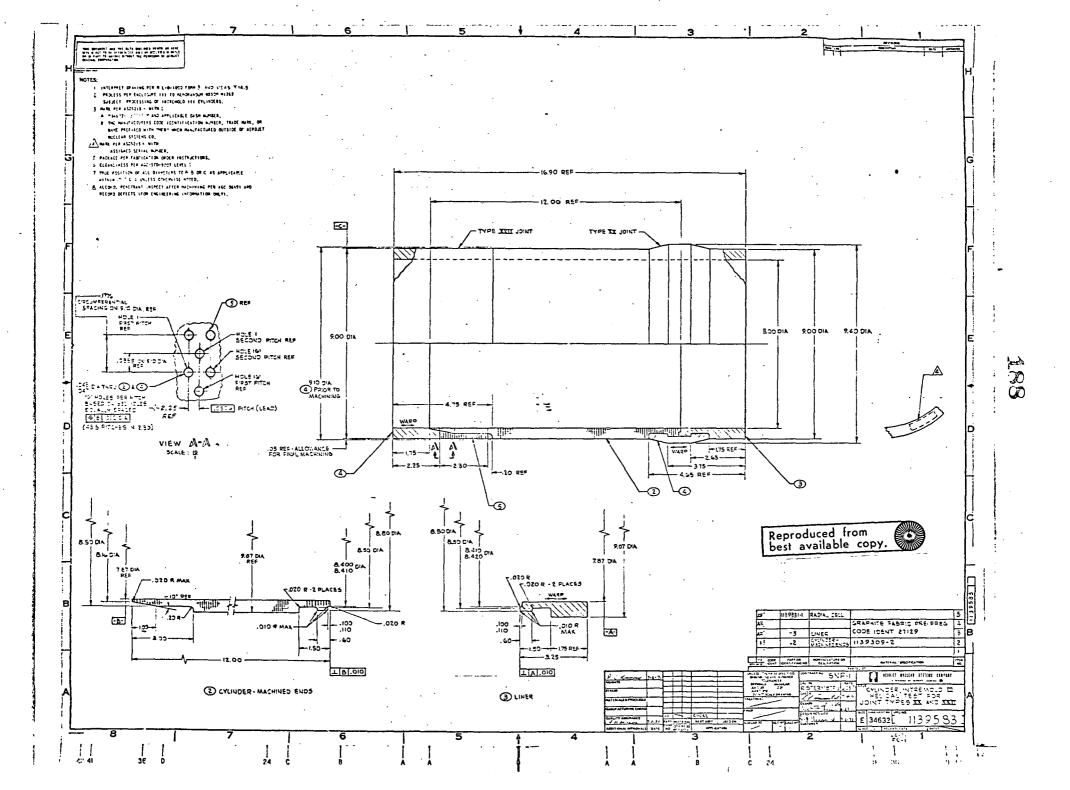


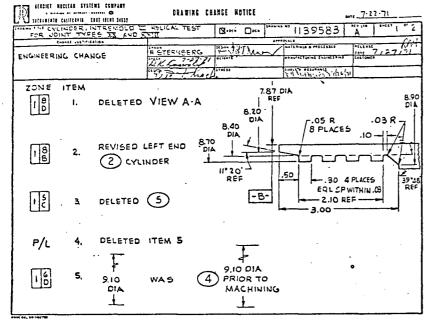


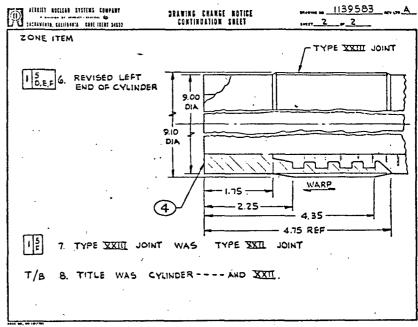












APPENDIX D

DEFINITION OF SYMBOLS

APPENDIX D

Definition of Symbols

Symbol Symbol	Meaning
Α	Array (subscripted); cylinder OD surface area
A _c	Cell transverse area
A _i ; i = 1,2,3	Transverse area of cylindrical annulus
Ap	Transverse area of ply
A_U	Unit area on cylinder OD surface
A _T	Radial cell transverse area
С	Outer diameter, or cylinder; cell (subscripts) diameter
d _O	Cylinder diameter before lateral debulk
d	Cylinder diameter after lateral debulk
f _z	Axial contraction factor
F	Cumulative normal distribution function
f_{θ}	Hoop (circumferential) extension factor
f	Extension factor; fiber (subscript)
1	Cylinder length after lateral debulk
10	Cylinder length before lateral debulk
L	Radial cell length
m .	No. bond surfaces in a joint
n	No. filament layers in a joint
N	Sample size; number of cells/cylinder.
n _p	Number of plies/inch on mandrel surface

Symbol	Meaning
N_{P}	Total number of plies
p	Lateral debulked axial cell spacing
r	Resin; radial coordinate
ra	Area ratio; fraction of cylinder area on OD surface occupied by cells.
R .	Weight resin in cell weight cell
S	Shear (subscript); blunting factor; spool (subscript)
\$	Lateral debulked circumferential cell spacing; sample standard deviation
t	Ply thickness; thickness; standard normal random variable
T	Transverse; total (subscripts)
u	Unit (subscript)
W	Weight; wound (subscript)
V	Volume
X	Sample mean
X _i ; i = 1,2,3	z - axis coordinate of joint feature transferred to ply
Y_{i} , $i = 1,2,3$	Length along ply surface in θ direction
z	Axial cylindrical coordinate
β	(Beta) Initial, or as-wound helix angle
β ₂	Final, or as-debulked, helix angle
ρ	Density
θ	(Theta) circumferential cylindrical coordinate

APPENDIX E

QUALITATIVE COMPARISON OF JOINTS

APPENDIX E

QUALITATIVE COMPARISON OF JOINTS

Table 28 ranks the joints according to the joint strength estimate, ease of manufacture (including scale-up and reproducability), and cost (where a rank of 1 corresponds to the lowest cost). Table 29 lists the joints in descending order of total ranking (last column of Table 28).

TABLE 28

JOINT RATING

<u>Joint</u>	Strength <u>Estimate</u> Rank	Ease of <u>Manufacture*</u> Rank	<u>Cost</u> Rank	Total
Thread Lock	2	- 6	6	14
Buck Tooth	3	2	2	7
Butt Clamp	8	5	5	18
Comb Tooth	1	9	9	19
Interlock	2	1	1	4
Straight Lap	7	4	4	15
Ledge Lap	4	9 .	6	19
Washboard Lap	6	3	3	12
Wave Lap	5	3	3	11

^{*} Includes scale-up estimate and reproducability.

TABLE 29

JOINT RATING SUMMARY

Place	Joint	Score
1.	Interlock	4
2.	Buck Tooth	7
3.	Wave Lap	11
4.	Washboard Lap	12
5.	Thread Lock	14
6,	Straight Lap	15
7,	Butt Clamp	18
8,	Ledge Lap	19 ~ Better in two out of three categories than Comb Tooth
9,	Comb Tooth	19

APPENDIX F

PROCESS SUMMARY CHART

PART B

FINAL REPORT ON CARBONIZING/GRAPHITIZING
PROCESSED OF SIX AGCARB/INTREMOLD III CYLINDERS

REPORT NO. ME-72-125

Final Report

FINAL REPORT ON PROCESSING OF SIX AGCarb/INTREMOLD III CYLINDERS

 $\mathbf{B}\mathbf{y}$

H. O. DAVIS

Aerojet Liquid Rocket Co.

Chemistry and Materials Design and Analysis Department Sacramento, California

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REFERENCES

Ref. No. Description Thacher, E. F., "The Manufacture Through First Cure of Six Experimental Intremold III Cylinder Assemblies" l. ANSC Report N8500, 72-R006, Jan. 21, 1972. Davis H. O./Brown J. A. to L. M. Swope "Nozzle Extension-2. Documentation of Dimensional and Weight Change Data for AGCarb Liner/Intremold III Cylinders" Aerojet Liquid Rocket Company, Materials Engineering Department Report No. ME-71-162 Dec. 9, 1971. Davis H. O./Brown J. A. "Nozzle Extension- Documentation 3. of Carbon Yield and Other Properties of Candidate Binder Systems for AGCarb Composites Aerojet Liquid Rocket Company, Materials Engineering Department Report ME-72-122, April 1972. 4. Crow, Edwin L., et al, Statistics Manual Dover Publications, Inc. New York, 1960.

I. <u>INTRODUCTION</u>

A. BACKGROUND

AGCarb 101, a two-dimensional (2D) reinforced laminated fibrous graphite composite was proposed for use as the material for fabricating the Nerva nozzle extension, due to its excellent combination of high temperature properties. However, in the flange attachment region the calculated stress level of 2500 psi is about double the shear strength level of the 2D composite. A higher cross ply tensile strength than available in the 2D AGCarb was also required. Intremold III, a three-dimensional composite was selected to provide the required interlaminar shear strength and generally more isotropic properties in the attachment flange. This approach necessitated development of a 3D to 2D composite joint. Carbon-to-carbon bonding of the two materials and integral mechanical interlocking were two techniques investigated.

The joining of one fibrous graphite composite to another to obtain optimum strengths at the interfaces required investigation of different joint designs. A total of 12 designs were made. Six cylinders approximately 8-in. dia, x 16 to 18 in. in length and approximately 0.5 in. wall thickness were fabricated. Each of the six cylinders contained a center section of Intremold III with laminated 2-D AGCarb design on either end, utilizing rosette (spiral) and shingle layup fabrication. The design, details of construction and processing up to and including first graphitization are contained in references 1 and 2.

All materials (the raw materials are listed below) and treatments of materials utilized in fabrication of the AGCarb/Intremold III cylinders are applicable to fabrication of a full scale Nerva nozzle extension.

1. AGCarb

AGCarb is the Aerojet-General Corporation designation for fiber-reinforced carbonaceous matrix composite materials fabricated in accordance with processes specified by AGC. Although there are variations depending upon process parameters, AGCarb consists of a graphite fabric, WCA from Union Carbide, impregnated with a carbonaceous filled USP 39 resin from U.S. Polymeric Chemicals Incorporated.

2. Intremold III

Intremold III used in this investigation contained WYB 125, Union Carbide graphite yarn and UPP 39 resin. Three helically and three orthogonally wound cylinders were made. These starting materials received the layup, cure, carbonization and graphitization related in reference 1 and 2, plus the additional densification and stabilization processing covered in this report.

3. Densification Binder

The densification binder (impregnant) used to reimpregnate the cylinders of AGCarb/Intremold III was a mixture of 15V carbon pitch and Indene. Indene C₉H₉ is a product of Henley Corp. and 15V is a product of Allied Chemical Corporation. The reinforcements, resins and binder plus the processing covered in this report identify a unique "AGCarb" composite. To date a number identification has not been assigned.

B. SCOPE OF INVESTIGATION

The data presented in this report was taken from the process history and physical changes of the AGCarb/Intremold III cylinders after the first graphitization through the final graphitization. A flow chart of the complete history of process steps is shown in Figure 1.

Of the processing steps shown in Figure 1, four reimpregnation (densification) steps, four cure cycles, four carbonization cycles and a final graphitization cycle are covered in this report. The source for the summarized data was the Process Identification Number (PIN) log and the Process Definition Documents (PDD). These documents, at present, are on file in individual fabrication history logs with ANSC Quality Assurance.

The dimensional measurements on the six cylinders identified as PIN 2001, 2002, 2003, 2004, 2005 and 2006 were taken at the same location as reported in Reference 2. The same template patterns were used for locating reported I.D., O.D., and length measurements. Processing information, including viscosity, specific gravity, and temperature of pitch were taken from the laboratory data sheets. Mixture ratios, additions to the original mix, evacuation times, vacuum readings, and pressure/time cycles were also recorded on the laboratory data sheets. All of these sheets are filed by PIN in the fabrication history logs.

II. SUMMARY/CONCLUSIONS

A. SUMMARY

The process history and physical changes occurring during processing of six AGCarb Liner/Intremold III cylinders for the NERVA SNP-1 Program were documented and recorded in process history logs. The cylinder dimensions were approximately & inch inside diameter, 0.5 inch thick wall, and 16 inch length. They contained Intremold III helically or orthogonally wound center sections integrally joined to AGCarb (two dimensional) rosette or shingle lay-up construction sections. Twelve transition joint designs were used to join the AGCarb to the Intremold III sections. Construction and processing through first graphitization is discussed is References 1 and 2. This report includes the documentation from initial graphitization through final regraphitization and covers the process steps impregnation, cure, carbonization and regraphitization.

Each cylinder was impregnated, cured and carbonized four times. All cylinders received a final regraphitization at 5000°F. A mixture of 15V/ Indene was used as the impregnant (binder). The mixture ratio was 75 percent by weight 15-V carbon pitch and 25 percent by weight Indene (C₉H₉). Indene, a clear, light liquid, was used as a solvent and to reduce the viscosity of the impregnant mixture. After evacuation to 27" Hg minimum, the cylinders were pressure impregnated at 200-225 psi. The pitch was cured at temperatures up to 500°F in circulating air and carbonized up to 1550°F in a flowing argon atmosphere. Graphitization up to 5000°F was also accomplished in an argon atmosphere. The six cylinders were processed in two groups of three each.

Group 1 included cylinders 2001, 2002, and 2003 and Group 2 included cylinders 2004, 2005, and 2006. Group 2 cylinders received more uniform processing which was reflected in reduced variability in weight pick-up and carbon yield. Weight and dimensional changes were recorded upon completion of each significant step in the processing.

The process variables of impregnant mixture viscosity, specific gravity, and temperature, before and after impregnation, were evaluated in relation to percent weight pick-up, volatile loss and final carbon yield. Linear regression analyses were made to show the percent of variable "y" explained by variable "x". In general the cause/effect relationships were as expected. Some relationships appeared to be non-linear but were not analyzed as such. Correlation coefficients, r, were calculated and many exceeded the critical absolute values at the 95% significance level. Various combinations of variables had high correlation coefficients just below the 95% significance level, indicative of other relationships which could be used for predicting process effects relating to impregnating fibrous graphite composites. Significant negative correlations were observed between cylinder specific gravity and percent weight pick-up during impregnation. A specific gravity goal of 1.40 was established for the cylinders after regraphitization and was achieved with final specific gravities ranging from 1.40 to 1.45. Basic process variable effects were somewhat obscured by the variation in cylinder starting specific gravity. The difference in relative volumes of AGCarb and Intremold fibrous graphite within individual cylinders contributed some difficulty in explaining weight pick-up and carbon yield variability. Variables which had measurable effects

on cylinder properties were viscosity, temperature, evacuation time, removal temperature, and technique for removing excess pitch. Location in the cure oven was also a variable with cylinders near the hotter oven walls tending to bubble or blister more than those centrally located.

Cylinder O.D., I.D. and length shrinkage was, with few exceptions, less than 1% from the initial cured through final regraphitization cycles. The orthogonally wound cylinders shrank more on the O.D. and had less increase in inside diameter than the helically wound cylinders. The dimensional changes in the helical cylinders is believed to be a function of matrix shrinkage and scissoring of the helix yarns while the movement of the orthogonal cylinders is primarily a function of resin shrinkage. As expected, the length of the helically wound cylinders decreased more than the orthogonally wound cylinders. Although the precision of the dimensional measurements was not high, the overall change in cylinder dimensions tended to be in the same general direction between graphitization and regraphitization cycles. The fibrous graphite was not totally stable after initial graphitization. Small changes in cylinder O.D., I.D., and length were recorded between graphitization and regraphitization.

Cylinder dimensional changes induced no delaminations or separations which could be detected by alcohol penetrant tests or radiographic inspection. The overlay hoop windings of PIN 2001 did delaminate, but the delaminations were in a circumferential filament wound area of a joint and not in the AGCarb liner or Intremold III. The Intremold III sections were readily permeable to isopropyl alcohol while the AGCarb rosette and shingle sections were not.

B. CONCLUSIONS

- 1. The specific gravity goal of 1.40 minimum for the AGCarb Liner/Intremold III combinations was achieved with four pressure impregnations of 15V/Indene.
- 2. The changes in length, O.D. and I.D. dimensions for the AGCarb Liner/Intremold III cylinders were less than one percent from as-cured dimensions to regraphitization dimensions.
- 3. The differential strain (resulting from shrinkage) between AGCarb Liner and Intremold III produced no visible separations or breaking of the carbon to carbon bonding between the two materials.
- 4. The helically wound cylinders tended to increase in outside diameter more than the orthogonally wound cylinders.
- 5. The net change in wall thickness was greater for the AGCarb Liner material than for the Intremold III material.
- 6. The AGCarb rosette construction shrinks more on the O.D. than either the AGCarb shingle, Intremold III helical or Intremold III orthogonal constructions.
- 7. It was demonstrated that two types of fibrous graphite construction can be integrated in structural shapes in the uncured state and subsequently processed to produce fully graphitized, dense components. Strengths of transition joints between differing basic construction types have not been determined to date.

- 8. The carbon yield of the 15V/Indene 75/25 impregnant mixture is approximately 40 percent, but varied from 20 to 78 in the cylinders.
- 9. The predicting equations and/or regression relationships between variables derived for the subject cylinders apply generally to the impregnation of fibrous graphite; however, curve slopes and/or constants would be different for structures produced using only AGCarb Liner or Intremold III constructions.
- 10. Processing results and relationships generally followed expected trends; however, overall tighter control of processing limits produced more uniform results.

III. TECHNICAL DISCUSSION

A. DENSIFICATION BINDER (IMPREGNANT)

A mixture ratio of 75/25 15V/Indene was used to impregnate the six cylinders. The selection was made based on the results of the binder investigation of the Nerva Program, Contract SNP-1, M-6 Plan.

The 15-V pitch is characterized as follows:

Softeni	ng Point	90 - 95°C
		(194 - 203°F)
Specifi	c Gravity (25°C)	1.26 - 1.32
Coking	Value	
	7 min. 950°C	26% min.
	10 min. 700°C	30% min.
Ash Per	cent	0.25% max.

Indene (C_9H_9) is a clear light liquid. The average assay of the Indene technical is 90-92% pure. The viscosity at room temperature is less than 1 when measured on the Brookfield viscosimeter #1 spindle at 10 rpm. It readily mixes with the 15-V pitch after the pitch is liquid; however, initial mixing is enhanced by agitation.

Viscosity, specific gravity and carbon yield data are given for 15-V Pitch and 15-V Pitch and Indene mixtures in reference 3. However, the viscosity vs. temperature curves shown in Ref. 3 do not show the effects of aging and additions of new mix and Indene. The 15-V/Indene mixtures used for the impregnation of the AGCarb/Intremold III cylinders were used up to five times. Each time the impregnant was exposed to heat up to 200 to 300°F for periods of 4 to 12 hours. In addition the mixture was exposed to preheat temperatures of approximately 200°F for 12 to 16 hours (overnight warmup). Addition of both fresh pitch and Indene were made as needed to replace mixture lost in impregnation and from evaporation during the heating and evacuation cycles.

The characteristic aging effects for the 15-V/Indene mixture after the third impregnation is shown in Figure 2. The viscosity as a function of temperature from 180°F to 280°F is shown for the mix prior to impregnation and after impregnation. Viscosity differences are much greater at the lower temperatures. For example, the <u>before</u> viscosity at 200°F is 40 to 50 cps and the <u>after</u> viscosity is 1000 cps at 200°F. Based on the changes in viscosity and specific gravity, it appears that aging consists of primarily a loss of Indene and some low molecular weight hydrocarbons.

The amount or degree of polymerization was not determined. The carbon yield after 5 impregnations was approximately 40 percent. The same yield was obtained from a fresh mixture of 15V/Indene. A high degree of polymerization would be expected to increase the carbon yield. The molecular weight distribution of the new batch vs. the aged batch would possibly be different at the same viscosity. The effects of aging are compounded by the additions of 15V and Indene.

B. PROCESSING - IMPREGNATION, CURE, CARBONIZATION AND REGRAPHITIZATION

Each of the cylinders was processed four times through the impregnation, cure and carbonization. The impregnation was accomplished in a sinterseal impregnator, serial No. B-1055 manufactured by National Sinter-Seal Div.,

N.B. Newcomb Co., Inc. Cure was in an air circulating oven and carbonization

was accomplished in a welded retort in a resistance heated furnace. The

cylinders were impregnated individually, but were cured and carbonized in groups

of three. PIN 2001, 2002, 2003 were processed together as the first group.

PIN's 2004, 2005 and 2006 were the second group. After the fourth carbonization

the cylinders were regraphitized to complete the process.

1. Impregnation Processing

The impregnation process was accomplished within the following parameters and limits.

a. Mixture Ratio (15-V-75% by weight/Indene-25% by weight)

Additions to the mixture were made in the same ratio;
however, if viscosity was above 50 cps at 250 ± 25°F, Indene was added to lower viscosity below 50 cps. Viscosity was measured at temperature with Brookfield

Viscosimeter RVF #1 Spindle at 10 rpm. Specific gravity was not used as a controlling parameter; however, it was measured with a hydrometer at approximately the same temperature that the viscosity was measured.

b. Heating Cycle

The mixture was heated overnight in an air circulating oven with a vent. The oven was set at approximately 200°F for overnight heating and raised to 300°F two to three hours prior to planned use of the pitch mixture. Viscosity and temperature were checked and additions made until temperature was between 225 to 275°F and viscosity was below 50 cps.

and viscosity, it was placed in a metal container in the impregnator. (In some instances, the heating was accomplished in the metal container.) The container was wrapped with a Ramaflex heater blanket type F-1479-1, Rama Corporation, San Jacinto, Calif. The blanket was insulated with asbestos wraps and taped to the container. Efforts were made to cover the heater blanket completely in case of pitch spillage or overflow. After the container was placed in the impregnator, the thermistor (control) was turned to the "On" position. The temperature controller for the blanket was set at 350 ± 25°F. For some of the final impregnations, the controller was set at a lower temperature or turned "Off" approximately 30 minutes prior to end of pressurization cycle. This was to reduce run-off and bubbling as the cylinders were removed from the impregnant. The changes and their effects are discussed in more detail below.

c. Preparation of Cylinder for Impregnation

The cylinders were weighed and measured prior to each impregnation. The data are recorded in Table I through VI under column headed weight of part.

The cylinders were impregnated individually; however, preparation for each followed approximately the same sequencing. After weighing the cylinders were attached with wire to the hydraulic vertical lowering ram in the lid of the impregnator. The extension of the ram and wire was checked each time to insure sufficient length for immersion of the cylinder into the mixture when the ram was fully extended. A metal weight was also attached to the cylinder to insure complete immersion.

The impregnator lid was closed and locked into position with the hydraulic arm in the retracted position. The cylinder was directly above the heated pitch mixture prior to starting the evacuation cycle so that it could be lowered into the solution after the evacuation procedure was completed.

d. Evacuation

With the cylinder above the hot pitch mixture, the impregnation chamber was evacuated from three to five minutes to a vacuum gage reading of 25 to 29 in. Hg.

The cylinder was then lowered into the pitch mixture. Vacuum was maintained for 0.5 to 5 minutes with the cylinder immersed in the pitch solution prior to pressurization.

e. Pressurization

After the cylinder was immersed, the impregnation chamber was pressurized with nitrogen to 200 to 225 psi. Pressure was maintained for 2 ± 0.25 hours. Pressure and temperature were checked approximately every 15 minutes until the end of the pressure/temperature cycle. At the end of cycle, pressure was released to one atmosphere and the impregnator was opened after raising the cylinder out of the pitch mixture.

2. Curing Cycle

Immediately after removal from the impregnator, and prior to cure the cylinder was wiped with a cloth to remove surface excess material. The cylinder was weighed and the value was recorded on the Impregnation History Sheet. The weight history for each cylinder and cycle have been summarized in Tables I through VI. The weight of cylinder and impregnant is shown in column WP+I. The weight of the impregnant, WI, and percent by weight of part pickup, % pI is also shown in Tables I through VI for each impregnation cycle.

The cylinders were reweighed after cure and this data appear in the column WP+CuI, weight of part plus cured impregnant weight. The volatiles (weight loss) and the cured impregnant weight are also shown. Percentage figures are also given for volatiles in Tables I through VI.

3. Carbonization

After weights were taken, the cylinders, in groups of three, were placed in a stainless steel (AISI 347) retort. Each cylinder was individually wrapped in graphite cloth and/or carbon felt. An inert atmosphere was

maintained during the carbonization cycle. Argon, 99.999% pure, was used. Chopped pieces of .045-in. dia. titanium (Ti 55A) wire getter was placed in the inlet tube of the retort prior to each carbonization cycle, to remove any moisture or oxygen in the argon.

The standard three-day carbonization cycle per ANSC Specification 90293, was used for converting the pitch to carbon. The cycle includes a six-hour hold at 1550°F. A flow of argon through the retort at 10 to 20 CFH is maintained during both the heatup and cooldown phase of the carbonization cycle. Cooldown rate did not exceed 100°F per hour. The instructions and precautions given to technicians for the carbonization cycle are listed in Appendix I.

After each carbonization the cylinders were weighed and measured. The weights are recorded in Tables I through VI. The percent carbon yield, cumulative percent carbon were calculated based on the weight of carbon and are shown in Tables I through VI for each of the four impregnations, cure, carbonization cycles.

The specific gravity after each carbonization was determined by water displacement and corrected for water absorbed by reweighing the wet cylinder immediately. The water absorb is subtracted from weight in $\rm H_2O$ prior to calculating specific gravity.

C. REGRAPHITIZATION

After the fourth and final carbonization, each of the cylinders were regraphitized at 5000°F. The actual graphitization cycles for the cylinders are filed in the individual PIN folders. The weights after graphitization are

shown in Tables I through VI. A full set of I.D. and O.D. measurements at each section and position were taken after the regraphitization.

D. DISCUSSION OF RESULTS

The goal of the densification processing was to achieve a minimum of 1.40 sp. g. in as few impregnations as possible. Three conditions controlled the number of impregnations. They were (1) specific gravity of cylinder after first graphitization (2) percent pick-up of impregnant and (3) carbon yield of the impregnant. The first two are a function of the porosity of the cylinder. Items (2) and (3) are a function of the process variations. The porosity of the cylinders was not measured; therefore, the discussion of results and effects of variables is limited primarily to process variables.

1. Specific Gravity - Cylinder

The specific gravity for each cylinder after the first graphitization, after each carbonization and after regraphitization is shown in Tables I through VI. The beginning specific gravities were somewhat different for the six cylinders and the final (after regraphitization) specific gravities ranged between 1.40 and 1.46. The bar graph in Figure 3 show the comparative beginning and final specific gravities. In general, the second batch of cylinders showed a greater increase in specific gravity. The specific gravity results for PIN 2004, 2005 and 2006 are consistent with the more uniform processing of the second batch of cylinders --discussed below. However, to ascertain the specific gravity differences, if any, related to the porosity of the Intremold III sections, the percent by weight and volume of AGCarb in each cylinder would have to be evaluated as a possible contributor to the variation. The general qualitative differences in volume are shown in Figure 1, reference 1. For example

PIN 2006 and 2006 had the lowest starting density and they have the greater percent by volume of Intremold III based on the cross section lay-out.

After the cylinders are sectioned the differences in specific gravity of AGCarb and Intremold III can be readily ascertained.

2. Weight Pick-up of Impregnant

It was anticipated that percent weight pick-up of impregnant based on the weight of the cylinder should correlate negatively with the specific gravity. (See further discussion and correlation coefficients for this and other relations of process variables in Statistical Analysis.) The trend observed was a negative correlation, i.e. the lower specific gravity cylinders had higher percentage weight pick-up. Variations in drainage, blistering during cure also affected the pick-up percentages.

The percent weight pick-up by cylinder for each impregnation is shown in Figure 4. There was a large variation in percent pick-up between cylinders; however, the trend indicates a sharp decrease in percent pick-up after the first impregnation and a lesser subsequent decrease.

The second impregnation for PIN 2001 and the third impregnation for PIN 2003 did not follow the general trend for decreasing weight pick-up with each successive impregnation. The 2nd impregnation percent weight pick-up for PIN 2001 was lower than the 3rd. The reason for the low pick-up for the second impregnation could not be established.

The 3rd percent weight pick-up for 2003 was lower than the fourth. Ten pounds of Indene were added to the mix just prior to the 3rd impregnation of PIN 2003. These are two examples of the effect of variations in

weight pick up. Better control and uniformity were achieved for the 2nd batch of cylinders, PIN 2004, 2005 and 2006.

The following is a list of variables which were factors affecting percent weight pick-up. However, all were not quantitatively measured in this investigation. Those measured in this investigation are discussed further in the Statistical Analysis Section. Some have been isolated in other studies for the M-6 Program, reference 3.

- (1) Porosity of Cylinder (not quantitatively defined)
- (2) Evacuation time for cylinder above solution and immersed in solution
- (3) Viscosity and specific gravity of solution
- (4) Temperature of mixture viscosity and specific gravity when cylinder was removed from mixture.
- (5) Pressure when cylinder is removed from mixture
- (6) Specific Gravity of cylinder
- (7) Temperature of cylinder when excess is removed by cloth wiping
- (8) Technique for removing excess pitch

In addition the cured weight retained is significantly affected by the condensation of volatiles on the cylinders. (This was previously recorded for flat test panels in the M-6 program.) The condensation occurs during evacuation of the chamber prior to immersing the cylinder. If pitch was introduced from an outside reservoir after evacuation was completed, less time would be allowed for condensation to occur. For the second batch of cylinders the time

above solution was reduced as much as possible to reduce variation in cured weight pick up caused by condensation of Indene and other low molecular weight hydrocarbons on the cylinders.

3. Carbon Yield - Binder

In relation to number of impregnations required, the weight retained after each impregnation, cure and carbonization was the controlling variable. The weights retained (carbon yields) are shown in Table I through VI. In General the percent carbon yield based on weight of impregnant was higher for the initial impregnation. One exception was PIN 2001 which had a higher yield (based on weight of impregnant pick-up) for the 2nd impregnation. However, the pick-up was very low for the 2nd impregnation of PIN 2001. Impregnation number one for PIN 2002 and number three for PIN 2003 also had low weight pick-up and comparatively high carbon yield. As can be seen in Figure 5, the reproducibility for weight pick-up and carbon yield was much better for the 2nd set of cylinders PIN 2004, 2005, 2006 processed.

The erratic results for PIN 2001, 2002, and 2003, shown in Figure 5 are a result of several inconsistencies in processing. The cause-effect relationship cannot be individually assignable; however, the following differences for the 1st and 2nd batch of cylinders existed.

	lst Batch Pin 2001, 2002, 2003	2nd Batch Pin 2004, 2005, 2006					
Evacuation Time	, , ,	, .,					
a. Above solution -	up to 5 min.	up to 3.5 min.					
b. Immersed -	up to 5 min.	up to 0.5 min.					
Blanket Thermistor Setting	350°F	350°F					
Time Blanket on	2 hrs.	1-1/2 hrs.					
Pressure	200 - 225	200 - 225					
Temperature of Pitch after Cycle	up to 295°F	up to 250°F					
Method of Removing Excess Pitch	Wipe with cloth - some bleeding observed.	Wipe edges and surface lightly - very little bleeding observed.					
Cure Cycle in Oven	Open to direct hot air from oven.	Place Al foil between oven wall and cylinder					

These changes were made after reviewing the laboratory data sheets for the first batch of cylinders and as a result of observation made during the impregnation processing.

In addition to the variations discussed above there is a difference in percentage by weight and by volume of AGCarb and Intremold III between the various cylinders. The difference by weight is not measurable; however, the difference by volume can be seen in the cross section of Figure 1, reference 2. Furthermore, there were undetermined differences in the density of the Intremold III sections which contributed to variations in impregnant pick up as well as the initial and final specific gravity of the cylinders.

4. Impregnant Process Variations

In addition to the variations between cylinders and in the impregnation process technique; there were variations in the impregnant (binder) mixture. Before impregnation and after impregnation viscosity curves are shown in Figure 2 for 15V Pitch/Indene mixture. The curve for after impregnation is typical; however, the amounts of Indene added, amounts of fresh mix added, heating times and temperatures as well as number of times reused would affect the curve. The mixture after impregnation generally required additions of Indene and/or replacement 15V/Indene mixture. The viscosity curve in Figure 2 for the before mixture represents a fresh batch and the after curve represents a batch (plus additions) after three impregnations. Prior to reuse, Indene had to be added to bring the viscosity into the required limits, 40 - 50 cps. The viscosity over the temperature range shown in Figure 2 was not determined after each use; however, the temperature, viscosity and specific gravity for the mix prior to the impregnation of each cylinder is shown in Table VII. Two batches of 15V/Indene were used. The first batch of 15V/Indene was used five times (with additions). This included the 4 impregnations of 2001, 2002 and 2003 plus the first impregnation of PIN 2004, 2005 and 2006. The second batch was used three times, the 2nd, 3rd and 4th impregnations of PIN 2004, 2005 and 2006.

5. <u>Dimensional Changes of Cylinders</u>

The initial processing of the AGCarb Liner/Intremold III Cylinders was discussed in reference 1 and 2. Dimensional changes from Cure through Graphitization were recorded in reference 2. The major changes in

I.D., O.D. and length measurements occurred during the processing up to and including the first graphitization. However, a continual check on dimensional changes was made during the impregnation, cure, carbonization and a final regraphitization processing.

In general, shrinkage and/or distortion have been related to delaminations in fibrous graphite composites. The combination of AGCarb in rosette and shingle lay-up with Intremold III in both orthogonal and helically filament round cylinders was suspect for non-uniform dimensional changes even after first graphitization. Therefore, a large number of measurements were taken at the end of each carbonization or graphitization process cycle. Tables VIII, IX, X and XI are examples of the number of measurements taken for the outside diameters of each cylinder and the calculations made to ascertain the change in O.D. at each point. Tables VIII and IX contain measurements and calculations for PIN 2001 and Tables X and XI for PIN 2002. The location of measurements are shown in Reference 2 in Table VIII and Figure 1. The type of fibrous graphite structure at the specific location is also identified in the tables of this report for ease of reference.

The measurements and calculations were repeated for the remaining four cylinders, but are not included in this report. In addition, inside diameter measurements and changes were recorded and are on file with the process history logs. However, the percent change is recorded in this report for all measurements including length and these are discussed below.

Templates were used to assist in repeating the location of measurements. However, there was still some variation in location which could not be eliminated. In addition, a somewhat variable thickness in the carbonized pitch on the O.D. and I.D. contributed to measurement error.

There were trends observed in the diametral changes related to the material and/or type of construction. It is believed that a mandrel is necessary to prevent excessive movement and control dimensions, particularly ovality. Use of the graphite mandrels, reference 2, for post cure, first carbonization, graphitization and regraphitization, reduced the changes which would have occurred if the cylinders had been freestanding during these process steps.

Primarily comparisons are made for cylinders and construction for the following conditions.

- Original cure measurements versus final (regraphitization) measurements
- Fourth impregnation carbonization measurements versus regraphitization measurements
- First graphitization and regraphitization

a. Outside Diameters

The percent changes between the following process steps are shown in Table XII, XIII, XIV, XV, XVI, and XVII for cylinders PIN 2001, 2002, 2003, 2004, 2005 and 2006 respectively:

	From Completion of	To Completion of
(1)	First graphitization	Carbonization after 2nd Impregnation
(2)	Carbonization after 2nd Impregnation	Carbonization after 3rd Impregnation
(3)	Carbonization after 3rd Impregnation .	Carbonization after 4th Impregnation
(4)	First Graphitization	Carbonization after 4th Impregnation
(5)	Carbonization after 4th Impregnation	Regraphitization
(6)	First Graphitization	Regraphitization
(7)	Original Cure	Regraphitization

For the first three comparisons only one diameter at each section was compared. For comparisons (4) through (7) above, four diameters at each section were compared.

With very few exceptions, the percent change was less than <u>one</u> percent. Overall the percent changes were rather uniform regardless of location or section of the cylinder being measured. Furthermore, there were only small differences between cylinders.

Both shrinkage and expansion occurred as evidenced by both negative and positive percent change, (the limitations of measurements are discussed above). Some of the measurements indicate out-of-roundness or a change in roundness; however, the outside surfaces were rough. If any distortion occurred, it was less than one percent of the diameter.

(1) Graphitization to Regraphitization

Of primary interest was the change from Graphitization to Regraphitization. As shown by Figure 6, the Helix cylinders 2001, 2003 and 2005 increased in diameter more than the orthogonal cylinders 2002, 2004 and 2005 between original graphitization and regraphitization. The movement of the AGCarb liner material on either end of the Intremold III tended to move in the same general direction. For example, the orthogonal cylinders had more negative percent Δ O.D.'s than the helix cylinders.

The etiology of this phenomenon is not certain.

The shrinkage which does occur is in the pitch, matrix. Strain produced by this shrinkage will be a result of stress from the matrix material. The matrix material bonds or adheres to the reinforcement if it is exposed, but more likely to the carbonaceous matrix already present. The helix wound cylinders are less restricted in the radial and axial direction because of the scissoring action of the helically wound yarns. This movement can take place without observable damage to the composite. However, any movement possibly damages the matrix and/or matrix to reinforcement bond. An increase in diameter should have produced a decrease in length if scissoring occurred unless there was slippage at the joints. The change in lengths is discussed in more detail later. Briefly PIN 2005 showed a comparatively large reduction in length. PIN 2001 had practically no shrinkage. The length measurements for 2003 were either misplaced or not taken after first graphitization.

(2) Original Cure to Regraphitization

The comparison of percent change in outside diameters from cure to regraphitization is somewhat arbitrary because of the pitch retained on the outside surfaces of the cylinders. There were both increases and decreases in outside diameters of the cylinders. The percent change and average at each location is shown in Tables XII through XVII. The average change for the four positions at each section is shown as a bar graph in Figure 7. Again the orthogonal cylinders showed less increase in diameter. All average percent changes were negative with the exception of Section B in 2002 and Section C in 2006. Section C had both positive and negative changes; therefore the average of +.01% is not meaningful. Section B of 2002, +.21% is in a rosette/Intremold joint area.

The trend for orthogonal cylinders is for a decrease in outside diameter; however, the maximum average decrease was -.17%. The maximum shrinkage in O.D. for the AGCarb material was -.88 percent in the A Section of 2004. Section A is a rosette section.

Helically wound sections increased in diameter up to 0.86 percent for cylinder 2003. As stated above, the helix cylinders can scissor around the radial pins to relieve shrinkage stress. The orthogonal cylinders are more restrained and shrinkage occurs in wall thickness with a resulting decrease in outside diameter.

b. Inside Diameters

The measurements after process step completion are the same for inside diameters as for outside diameters listed above. The percent change for the six cylinders are listed in Table XVIII through XXIII. The

percent changes from first graphitization through fourth (final) carbonization were negative in almost every instance. This decrease in diameter is mostly from pitch buildup on the I.D. surface and does not represent any significant movement of the cylinders.

(1) Graphitization to Regraphitization

There were both increases and decreases in inside diameters. The percent changes tended to be more positive for helically wound cylinders indicating larger increases in inside diameters. The same was true for the outside diameters of helix cylinders discussed above. The helically wound cylinders are freer to move because of the possibility of scissoring of the hoop yarns between the radial pins. Again there was neither increase nor decrease greater than one percent.

The average percent change for each section of the cylinders including the joints, Intremold III and AGCarb sections are shown in Figure 8. In general a decrease in inside diameter corresponded to a decrease in outside diameter; however, there were many contradictions.

(2) Original Cure to Regraphitization

Figure 9 shows the average percent change from original cure dimensions to regraphitization for all sections of the six cylinders. The values are also listed in Tables XVIII through XXIII. With three exceptions all percent changes were positive with increases in inside diameter up to .84 percent. The three sections with negative percent change were from cylinder 2004 in the AGCarb rosette or AGCarb rosette/orthogonal Intremold III sections. These values are within 0.10 percent.

The pattern trends for movement of helix versus orthogonal were again apparent. The helically wound cylinders showed greater increases in percent change than did the orthogonal wound cylinders. Secondly the greater increase in percent change of inside diameters corresponded to more positive percent increases in outside diameters. Again, there are many contradictions.

c. Change in Length

The percent change in length at four locations, 0°, 90°, 180° and 270°, for all the cylinders are shown in Table XXIV. The length measurements for (1) original cure (2) first graphitization (3) 4th impregnation carbonization and (4) regraphitization are recorded and percent change between process steps was calculated from the recorded values. The average changes were negative showing a decrease in length between process step except for from "fourth impregnation carbonization" to "regraphitization". For this cycle PINs 2001, 2002, 2003 and 2006 were positive.

These increases in length between fourth impregnation carbonization, although small, are opposite from the expected. Additional shrinkage occurs during graphitization of the carbonized pitch matrix; therefore a decrease in length was anticipated. Cylinder 2005 did shrink as expected; and the average percent change was a negative .15 percent.

All cylinders had a decrease in length from "original cure" to "regraphitization"; however, cylinder 2005, a helically wound cylinder had the largest decrease of -0.92 percent. The next largest decrease was also a helix cylinder. PIN 2001 had a decrease of -.29 percent. Results from flat

plates of AGCarb liner material show practically no change in length or width; therefore, it is assumed that the changes in lengths are primarily a function of the Intremold III. The helical wound cylinders would be expected to experience greater length changes than orthogonal cylinders. PIN 2003 was an exception.

d. Change in Wall Thickness

The change in wall thickness was not measured; however, a review of the I.D. and O.D. measurements show a decrease in wall thickness with few exceptions. The wall thickness of PIN 2003 in the Intremold III helical wound section apparently increased in thickness. PIN 2005 at Section D, helically wound, also had a slight increase in wall thickness. Changes in wall thickness of 1 to 4 percent have been measured on flat plates of AGCarb. Based on the comparative O.D. and I.D. measurements it appears that changes greater than 4% and up to 10% may have occurred for the cylinders.

e. Inspection

Each cylinder was alcohol penetrant inspected after regraphitization.

Cylinders had been previously checked after Post Cure, Carbonization and Graphitization. Results are recorded in PIN Process History logs. Briefly PIN 2006 had delaminations for almost 360° around the cylinder .050 to .130 from I.D. after original graphitication in the IM shingle section. The depth could not be ascertained. The AGCarb liner Joint Design XVI of PIN 2006 was damaged on the sharp edge after carbonization to a depth of approximately .250" from inside diameter extending approximately 3".

Both the delamination and damage were sealed with pitch and/or removed during subsequent machining. A small indication from the damaged area in the rosette section is still visible. However, with this exception there were no delaminations or separations observed during alcohol penetrant check except on the circumferential filament windings of PIN 2001. The results of radiographic inspection were also negative as to delaminations and/or separations. There were; however, some questionalbe areas. Radiographic prints are on file.

The Intremold sections were porous. In an attempt to compare the relative porosity, alcohol permeability tests were conducted. The time for 10 cc of isopropyl alcohol to penetrate from I.D. to 0.D. was the gage used for comparison of Intremold and AGCarb sections. The results are contained in Table XXV. There was no observable penetration of the AGCarb with 10 cc of isopropyl alcohol after 4th impregnation carbonization or after regraphitization. There was no penetration of the Intremold III material of cylinders 2004, 2005 and 2006 after fourth impregnation carbonization. (PIN 2001, 2002 and 2003 were not checked). On the other hand all Intremold III sections were penetrable by isopropyl alcohol after regraphitization. The time varied from 5 seconds to 30 seconds.

PIN 2006 orthogonal and PIN 2003 were the least permeable. That is, more time was required for the alcohol to appear on the outside of the cylinder. These cylinders also had a greater spread in times to penetrate than the other cylinders. It is anticipated that these results would have related to specific gravity of the Intremold III. Since the cylinders were not tested this could not be verified.

f. Machining

After regraphitization and final measurements were taken, the cylinders were machined in accordance with applicable drawing. Some modifications were made to fit parts as fabricated. The nominal wall thickness was approximately .400 - .500.

The cylinders were machined with standard carbide cutting tools on an engine lathe. The cylinders were held to make molds for machining the outside diameters. They were chucked on O.D. to machine to inside diamensions. Each cylinder was faced to length with the carbide cutting tool. There were no machining discrepancies noted and apparently no damage to Intremold III or AGCarb material.

E. STATISTICAL AND GRAPHITICAL ANALYSIS

A statistical analysis of some of the measured cylinder data and process variables was conducted. Statistical analysis was used to verify and/or show which variables could be expressed as a function of another variable. These results were not treated rigorously, but used for trend indications and to show cause/effect relationships. After completion of the statistical analysis, additional graphs were made to depict the relationship between variables. The graphs and statistical analysis are supplemental to the preceding discussion of the results.

Primarily Linear Regression analysis was used in comparing variable "y" with another variable "x"; however, selected variables were analyzed using

Multiple Linear Regression analysis. For linear regression analysis those combinations which would be expected to interact were analyzed and the correlation coefficient r was calculated. Most of relationships identified were obvious and regression of variable "y" upon variable "x" was expected. A 5% significance level was used to test the computed values of r. If the computed value of r exceeded the critical value from Table 7, reference 4, the null hypothesis that the population of "x's" and "y's" had zero correlation was rejected. However, some of the correlation coefficients which were lower than the critical value at the 95% significance level show trends and should be investigated further.

The line of best fit was not computed for the linear regression lines shown in the graphs but were estimated from the plotted data points.

The results of both the Linear Regression and Multiple Linear Regression analysis are discussed in the following paragraphs.

1. Linear Regression Analysis

a. Correlation Matrix #1

 $\begin{tabular}{ll} The computed correlation coefficients r are shown in \\ Figure 10 for the following variables: \\ \end{tabular}$

- (1) Starting specific gravity of cylinder
- (2) Final specific gravity of cylinder
- (3) Percent weight pick up first impregnation based on weight of cylinder
- (4) First carbonization specific gravity
- (5) Second carbonization specific gravity

- (6) Third carbonization specific gravity
- (7) Fourth carbonization specific gravity
- (8) Percent weight pick up second impregnation based on weight of cylinder
- (9) Percent weight pick up third impregnation based on weight of cylinder
- (10) Percent weight pick up fourth impregnation based on weight of cylinder

All combinations compared have correlation coefficients r listed in the correlation matrix, Figure 10. The correlation matrix is symmetric; however, each correlation coefficient has been listed only once. Those combinations without correlation coefficient in the matrix were not compared. After reviewing the data, the computation for the combinations not shown were not considered necessary.

There were no significant positive correlation coefficients for the combinations "tested" in Figure 10. A positive $\, r \,$ indicates that the "y" variable increases with an increase in the "x" variable.

The significant negative correlation coefficients were between the following cylinder properties and/or process parameters.

- (1) Starting Specific Gravity of Cylinder vs. Percent weight pick up first impregnation based on weight of cylinder.
- (2) Starting Specific Gravity of Cylinder vs. First Carbonization Specific Gravity.
- (3) Starting Specific Gravity of Cylinder vs. Second Carbonization Specific Gravity
- (4) Starting Specific Gravity of Cylinder vs. Percent Weight pick up second impregnation based weight of cylinder.

(5) Percent weight pick up first impregnation based on weight of cylinder vs. first carbonization specific gravity, (cause for this relationship is a third variable-starting specific gravity of cylinder).

The variable listed on the left is most likely the causal variable.

There were other trends indicated by the relatively high values for some of the computed correlation coefficients which did not exceed the selected critical values at the 95% significance level.

Some of these however, are merely related and the cause for both variations is due to a third variable or some variation not measured.

Of the variables measured and compared in correlation Matrix #1, the starting specific gfavity is of primary importance in its effect upon (1) percent pick-up as well as (2) specific gravity after further processing. An estimated regression line of specific gravity (initial) versus percent pick-up is shown in Figure 11. The first and second impregnations for the six cylinders are shown. The slope has decreased for the second impregnation.

Figure 12 shows the regression line for the percent pick-up (2nd impregnation) versus specific gravity after the first carbonization cycle. The correlation coefficient between these two variables was relatively high at -.633 and just below the critical value. The relationship is apparent and useful for predicting percent pick-up. However, the specific gravity represents a

combination of AGCarb Liner and Intremold III which have different specific gravities, pore size and pore distribution. A better relationship for specific gravity versus percent pick-up could be established for either material individually.

pick-up based on weight of part versus specific gravity prior to impregnation.

The scatter about the regression lines is much less for the 2nd group of cylinders. Again, this is indicative of the closer control of processing the second group of cylinders.

b. Correlation Matrix #2

The computer program used to determine correlation coefficients did not allow for all variables in one computation. The second set of variables compared is listed in the legend of Figure 14. Figure 14 also contains correlation Matrix #2. The sample size for #2 was 13; therefore, the critical absolute value at the 95% significance level was ±.533.

There was only one negative correlation coefficient which exceeded the -.533 value. There was a significant correlation (-.786) between temperature of impregnant after impregnation cycle and percent volatiles in impregnant. In other words, the lower temperature of impregnant at cylinder removal time produces a higher volatile content in the impregnant contained in the cylinder pores. It is evident that a relative "hot" pitch would evolve more volatiles upon exposure of the cylinder to the atmosphere. (Note: There was a time lapse between removal of cylinder from impregnant and weighing; therefore, the comparatively hot cylinders will lose more volatiles between

removal and weighing.) The individual data points are plotted in Figure 15 and a linear regression line is estimated for this relationship.

Cylinders removed at the lower temperatures lost more volatiles during cure. The viscosity after impregnation is also a factor in drainage and volatile loss. The optimum temperature for removal was not established; however, a temperature between 220°F and 260°F is desirable. Above 265°F, bubbling or blistering may occur as well as excessive drainage. Below 200°F, the excess pitch is difficult to remove.

Other correlation trends were indicated. The following combinations had relatively high negative correlation coefficients:

- (a) Viscosity of impregnant after impregnation cycle versus percent pick-up of impregnant based on weight of part prior to impregnation cycle. r = -.500
- (b) Temperature of impregnant after impregnation cycle versus viscosity of impregnant after impregnation cycle. r = -.434

The variable listed on the left in the above comparison is likely the causal factor.

A positive correlation coefficient of .627 was obtained for temperature of impregnant after impregnation cycle versus percent carbon yield based on weight of impregnant. This relationship is a corollary to the negative relationship between (temperature of impregnant after impregnation)

versus (Percent Volatiles in impregnant). That is, volatiles were lost if the cylinder was removed relatively hot; therefore, the pick-up weight recorded contained less volatiles and naturally had a higher carbon yield based on impregnant weight.

c. Correlation Matrix #3

The third set of variables analyzed are shown in the legend of Figure 16, Correlation Matrix #3. All of these variables also appeared in Correlation Matrix #2. They are repeated in this analysis with a larger sample size, n = 24. The sample size was smaller for Matrix #2 because the after removal process data was not recorded for all cycles. The following combinations had positive correlation coefficients which exceeded the critical value ($\frac{1}{2}$.404) at a 95% significance level:

- (1) Viscosity of impregnant prior to impregnation versus specific gravity of impregnant prior to impregnation cycle. r = .727
- (2) Percent carbon yield based on weight of <u>cured</u> impregnant versus percent carbon yield based on weight of impregnant. r = .907

The high correlation coefficient for combination (2) indicates that the differential loss of volatiles and pitch from drainage between cylinders was quite small. It is assumed that the approximately 20 percent unexplained variation is a result of differential drainage and/or volatile loss. The individual data points and line of best fit by group for this relation is shown in Figure 17.

The data represents two separate batches of 15V/Indene and many additions of Indene and/or pitch and Indene. In addition the surface area of impregnant exposed in relation to the volume of fresh impregnant is progressively more with each impregnation. There was also more impregnant (percent by weight) actually on the surface of the cylinders for the later impregnations. This was brushed off after cure and does not appear in the carbon yield calculations. The brushing contributes to the carbon yield variability. All data analyzed in Matrix #3 was treated the same regardless of the number of the impregnation cycle. The prior impregnations were treated as having zero effect. It is obvious from the data that the converse is true. Furthermore, less variation in the process variables analyzed in Matrix #3 would be expected for one batch of pitch without additions of Indene.

A significant negative correlation was obtained between percent volatiles in impregnant based on weight loss during cure versus percent carbon yield based on weight of impregnant. An r of -.530 was computed. The higher carbon yield was obtained from the impregnant with lower volatile content prior to starting cure. The relationship is shown graphically in Figure 18.

One additional linear regression analysis (not shown in the matrix) was made. Specific gravity of the cylinders from both groups prior to each impregnation was compared to percent pick-up of impregnant based on weight of part prior to impregnation cycle. As expected a significant negative correlation coefficient was obtained. The value was -.453, which exceeds

the \pm .404 at 95% confidence level, n=24. The variation for percent pick-up for the first batch of cylinders was affected by the wider process variations. Therefore, a much higher correlation coefficient would be expected with less process variation.

2. Multiple Linear Regression

A cursory analysis was made to ascertain if predicting equations could be found using from 2 to 4 independent processing variables. As expected, the viscosity of 15V/Indene mixture is predictable from the specific gravity of mixture and temperature of the mixture.

$$y = -977.967 + 958.627 A - .326 B$$

The regression estimate or predicting equation is as follows:

 $y = b_0 + b_1 A + b_2 B$

y = predicted viscosity in cps

 b_0 , $b_1 \& b_2$ = best estimates of constants

A = Specific Gravity

B = Temperature °F

The index of determination for the linear regression of the (viscosity) upon the variables of (specific gravity) and (temperature °F) is 0.697. This is significant at the 1% level. The equation could be refined and improved with the analysis of more data.

The average and standard deviation for viscosity, specific gravity and temperature of the 15V/Indene mixture for all impregnations is shown in Table VII. Values for each impregnation are also tabulated.

Other variables analyzed included (1) y-percent carbon yield as a function of the independent variables A - viscosity, B - specific gravity and C - temperature and (2) y - percent pick-up as a function of A - viscosity, B - specific gravity and C - temperature.

Without further search and calculation neither of the above equations could be used based on the Multiple Linear Regression analysis. It is likely that other relationships exist. A tightening of upper and lower limits for process variables, as well as more uniformity in the samples being impregnated would aid in establishing and verifying these relationships. More rigorous statistical analysis techniques would produce additional relationships for predicting equations. Collecting of additional data should be proceeded by a selection of an experiemental design of process variables and cylinder properties to reduce possible interaction effects.

As a part of the linear regression analysis, means, (\overline{X}) and standard deviations, (S.D.) for the major process variables and cylinder properties were computed. These are listed in Table XXVI. These values are presented to show the variation for a particular property and/or process.

F. RECOMMENDATIONS

- 1. The maximum width of fabric which can be used without producing wrinkles should be determined for rosette and shingle lay-ups on a full diameter cone.
- 2. In conjunction with the first recommendation a full diameter short (6") demonstration cone or cylinder should be fabricated and processed through the first graphitization cycle to verify the scalability of the shrinkage and distortion data contained in this report.
- 3. The data contained in this report, reference 1 and reference 2 should be correlated with the physical and mechanical properties of the cylinders including the strength of the various joint designs.
- 4. The AGCarb, transition joints and Intremold III should be cross sectioned, polished and examined at 100X magnification or greater to establish uniformity of the composites.
- 5. Additional studies of process variations should be conducted to establish optimum impregnation, cure and carbonization cycles.
- 6. A technique and/or facility should be devised for conducting the evacuation of the fibrous graphite component separate from the impregnant.

TABLE I

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGGARD LINER/INTERMOLD III CYLINDER PIN 2001
THROUGH FOUR IMPREDNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Cure and	Impregnation, Carbonization yele	Wt. of Part N-P	Wt. of Part + Impregnant W*P+I	Wt. of Impregnant	% Impregnant Based on Weight of Part %pI		Wt. of Cured Imprognant W-Cu-I	Wt., off Volts. W*V	Vols. in Impregnant	Wt. of Part + Carbon W•P+C	Wt. of Carbon W•C	Carbon Based on Part Wt.	Cum- ulative %pC	Carbon Besed on Impregnant	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) After Carb.	,1 —
F	irst	5000	6,630.0	1630	32.6	6407	11:07	223	13.7	5932	932	18.6	18.6	57.2	66.2	1.30	
s	econd	5909	63145	436	7.4	6300	391	45	10.3	6193	284	4.8	23.9	65.1	72.6	1.37	
. т	hird	6193	6995	802	13.0	6822	629	173	21.6	6509	316	5.1	30.2	39.4	65.4	1.142	
F	ourth .	6509	7109	600	9.2	6992	L 83	117	19.5	6728	219	3.4	34.6	36.5	45.3	1.454	
Regraphit	ization									6682			33.6			1.45	3 N'

LENEND

Symbols for weight change and percent weight change Table I through VI.

P = Cylinder (Part)

I = Impregnant (Binder)

Cu = Cured

V = Volatiles - Drainage during cure included in all volatile calculations

C = Carbon - Weight retained after carbonization cycle W - Weight in grams

%p = Percent based on cylinder weight

%i = Percent based on impregnant weight

Specific Gravity was determined after carbonization and graphitization by water displacement and corrected by subtracting weight of water pick-up from weight of cylinder in water.

TABLE II

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGGIARD LINER/INTERMOLD III CYLINDER PIN 2002
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	₩•P	W•P+I	W•I	%oI_	W•P+Cu•I	W•Cu•I	M•A	%1V	W-P+C	W•C	%pc_	Oum- ulative %pC	<u>zic</u>	Based on Impregnant Veight After Cure	Specific Gravity (Corrected) After Carb.
First	3846	1790	944	24.5	1708	862	82	8.7	£586	740	19.2	19.2	78.4	85.5	1.312
Second	145145	5028	183	10.6	1966	757	62	12.8	4801	256	5.6	24.9	53.0	60.8	1.351
Third	1801	5070	269	5.6	5000	199	269	74.0	4908	107	2.2	27.6	39.7	53.8	1.375
Fourth	1908	5380	և72	9.6	5241	333	139	29.4	5068	160	3.3	31.8	33.9	48.0	1.415
	•														•
Regrephitization									5030	•		30.8			1.400

TABLE III

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGGARD LINER/INTREXOLD III CYLINDER PIN 2003 THROUGH FUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	₩•₽	W-P+I	W•I	%pI_	W-P+Cu-I	W-Cu-I	W•V	ziv	W-P+C	W-C	%pC	Cum- ulative %pC	*ic	SC Based on Impregnant Weight After Cure	Specific Gravity (Scrrected) ¹ After Carb.
First	3301.0	1500.0	1199	36.3	4343	1075	157	13.1	3948	6և7	19.6	19.6	54.0	62.1	1.252
Second	3948	4705	757	19.2	4595	647	110	14.5	h296	348	8.8	30.1	46.0	53.8	1.369
Third	և296	1432	136	3.2	14105	109	27	19.9	4383	87	2.0	32.8	64.0	79.8	1.390
Fourth	4383	4885	502	11.5					1,538	, 155	3.5	37•5	30.9		1.142
Regraphitization					•				4531			37.2			1.415

CHANCES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGGRED LINER/INTREMOID III CYLINDER PIN 2001, THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Oure and Carbonization Cycle	К •Ъ	W•P•I	<u> W·I</u>	%pI	W-P+Cu-I	W-Cu-I	₩• ₹	Fiv	W-P+C	W-C	%pC	Cum- ulative %C	<u>\$10</u>	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) After Carb.
First	5101	6930	1829	35.9	6536	1435	394	21.5	6044	913	18.5	18.5	51.6	65.7	1.260
Second	6052	7238	1186	19.6	6883	831	355	29.9	6528	<u> 1</u> 76	7.9	28.0	10.1	57-3	1.351
Third	6528	7511	983	15.1	7117	589	394	40.1	6825 .	297	4.5	33.8	30.2	50.4	1.1,01
Fourth	6825	7555	730	10.1	7345	520	209.5	28.6	7050	225	3.3	38.2	30.8	h3.3	1.392
Regraphitization						•			6937			36.0			1.148

TABLE V

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGGRED LINER/INTREMOLD III CYLINDER PIN 2005
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Ours and Carbomization Cycle	, ₩•₽	W-P+I	W·I	%I_	W-P+Cu*I	W•Cu•I	₩•∇	ziv_	W-P+C	<u>w-c</u>	\$pC	Cum- ulative %pC	%ic_	AC Based on Impregnant Weight After Cure	Specific Gravity (Corrected) After Carb.
First	4744	6685	1941	40.9	6295	1551	390	20.1	5807	1068	22.4	22.4	54.8	68.5	1.241
Second	5807	7019	1212	20.9	6635	828	384	31.7	6280	473	8.1	32.4	39.0	57.1	1.329
Third	6280	7232	952	15.2	6911	631	321	33.7	6575	295	4.7	38.6	31.0	16.8	1.410
Fourth	6575	7342	767	11.7	7058	483	784	37.0	6745	110	2.6	42.2	22.2	35.2	1.419
•															
Regraphitization									6652			40.2			1.441

TABLE VI

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGGRED LINER/INTREMOID III CYLINDER PIN 2006
THROUGH FOUR IMPRECNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Empregnation, Cure and Carbonization Cycle	<u>W.P</u>	W-P+I	W•I	£оI_	W•P+Cu•I	<u>₩•Cu•I</u>	<u>₩•₩</u>	%1V	₩•P+C	W-C	%pC	Cum- ulative %nC	%1C	AC Based on Inpregnent Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
Pirst	4136	5950	1814	43.9	51.52	1316	498	27.5	5042	906	21.9	21.9	19.9	63.8	1.241
Second	2075	6201	1159	23.0	5770	728	. 431	37.2	5470	128	8.5	32.3	36.9	58.8	1.313
Third	51.7L	6500	1026	18.7	6055	581	445	43.4	5765	291	5.3	39.4	28.4	56.6	1.360
Fourth	5765	6615	850	14.7	6330	565	285	33.5	5941	176	3.1	43.7	20.7	31.2	1.380
Regraphitization									5875			75.0			1.1,26

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VISCOSITY, SPECIFIC GRAVITY AND TEMPERATURE OF IMPREGNANT PRIOR TO IMPREGNATION CYCLES °F

AGCarb/Intremold III Cylinders

Number of				
Impregnation	Pin	Viscosity	Specific	Temperature °F
Cycle	<u>No.</u>	<u>CPS</u>	Gravity	
FIRST	2001	48	1.144	225
	2002	41 .	1.154	265
	2003	48	1.152	250
	2004	52	1.160	250
	2005	41	1.162	260
	2006	56	1.164	250
SECOND	2001	43	1.151	265
	2002	49	1.154	250
	2003	55	1.160	285
	2004	29	1.140	235
	2005	28	1.143	230
	2006	39	1.140	250
THIRD	2001 2002 2003 2004 2005 2006	39 34 39 69 26 46	1.138 1.144 1.142 1.156 1.140	250 248 252 220 255 255
FOURTH	2001	44	1.162	265
	2002	56	1.162	258
	2003	43	1.166	262
	2004	23	1.139	265
	2005	55	1.156	235
	2006	26	1.138	270
	Avg.	42.875	1.151	252.083
	Standard Deviation		0.010	14.981

TABLE VIII

OUTSIDE DIAMETER MEASUREMENTS OF AGGAR'S LINER/INTREMOLD III CYLINDER PIN 2001

Post Process Step Measurement in Inches Outside Diameter 2nd 3rd Lith Location Material Orig. First Imp. Imp. Imp. Regraph. Section Position Carb. Carb. Carb. Cure Graph 8.810 Intremold III 8.814 8.845 Δ Dl 8.792 8.373 8.815 8.830 8.837 8.807 8.810 D2 Helix 8.801 8.809 8.805 8.773 8.827 8.795 8.793 8.812 D3 DL Dl FM 5228 9.158 9.115 9.141 9.134 9.125 9.142 В 9.11:0 D2 Shingle 9.148 9.130 9.138 D3 9.136 9.11.6 9.136 7.136 9.152 9.140 9.160 9.156 8.81.7 8.835 8.821 8.854 8.8836 3.855 1.813 C Dl Intremold III 8.826 8.829 8.820 8.870 8.812 8.879 D2 Helix D3 8.857 8.817 8.813 8.860 8.879 9.299 9.22h 9.232 9.225 D D1 Joint II 9.224 9.222 9.216 9.214 9.205 D2 Intremold III 9.222 D3 • FM 5228 9.234 9.240 9.230 9.237 DL Shingle 9.230 9.223 9.215 9.262 9.155 B D1 FM 5228 9.133 9.124 9.120 9.122 9.128 D2 9.154 9.119 9.149 Shingle 9.131 D3 D4 9.155 9.11:4 9.131 9.118 9-153 9.123 9.120 9.158

do

TAPLE IX

CHANGE IN OUTSIDE DIAMETERS OF AGGARD LINER/INTREMOLD III PIN 2001

		•	Changes in Outside Diameters Between Process Steps									
	Diameter tion Position	Material	O.D. Graph to Material 2nd Imp. Carb.	O.D. 2nd to 3rd Imp. Carb.	0.D 3rd to 4th Imp. Carb.	O.D. 1st Graph to Lth Imp. Carb.	0.D. 4th Imp. Carb. to Regraph.	0.D. 1st Graph to Regraph	O.D. Orig. Cure to Regraph			
Å	D1 D2 D3 D4	Intremold III Helix	+•001	+.001	005	003	+.035 +.0321 +.004 005	+.034 +.020 -+.010 +.012	+.0353 +.029 +.032 +.034			
В	D1. D2 D3 D4	FM 5228 Shingle	004	003	009	020	+.017 +.002 +.00h +.02h	003 +.010 +.004 +.014	016 008 012 012 00h			
c	D1 D2 D3 D1	Intremold III Helix	֥003	+.001	+. 006	+.010	+.034 +.024 +.014 +.073	+.044 +.067 +.010 +.036	+.023 +.014 +.036 +.025			
. .	D1 D2 D3 D4	Joint II Intremold III + FM 5228 Shingle	002	+•00h	~. 002	010	+.018 +.020 +.007 +.047	+.008 +.003 003 +.034	+.003 +.001 +.003 +.032			
B	D1 D2 D3 D4	FM 5228 Shingle	009	-•007	+.002	011	+.006 +.030 0313 +.038	005 +.018 026 +.035	C27 C05 C35 +-CC5			

OUTSIDE DIAMETER MEASUREMENTS OF AGCATH LINER/INTREMOLD III CYLINDER PIN 2002

				Post Process Ste					
Outside 1					2nd	3rd	4th		
Loca	ation		Orig.	First	Imp.	Imp.	Imp.		
Section	Position	Material	Cure	Graph	Carb.	Carb.	Cerb.	Regraph.	
A	pl	FM 5228	9.198	9.180	9.146	9.156	9.117	9.153	
	D2	Rosette	9.178	9.144			9.129	9.090	
	D3		9.210	9.180			9.148	9.755	
	D4		9.182	9.145			9.132	9.107	
В	DI	JTXVI	8.859	8.927	8.924	8.930	8.931	8.962	
	D2	FM 5228	8.920	8.911			8.898	8,896	
	D3	Rosette	8.941	8.929			8.911	8.930	
	D4	Intremold III Ortho	8.898	8.862			8.885	8.894	
С	DI	Intremold III	8.781	8.785	8.824	8.832	8.769	8.761	
•	D2	Ortho	8.848	8.824	•••-		8.829	8.819	-
	D3	012110	8.838	8.892			8.830	8.827	₹V
	D4		8.839	8.827			8.823	8.822	N A
D	D1	Intremold III	8.822	8.817	8.818	8.824	8.800	8,805	1
-	D2	Ortho	8.843	8,826	0,010	0.02	8.818	8.820	
	D3	011110	8.845	8.835			8.827	8.833	
	D4		8.851	8.835			8.833	8.837	
E	DI	Intremold III	8.841	8.842	8.340	8.848	8.8821	8.813	
	D2	Ortho	8.830	8.838			8.814	8.843	
	D3		8.838	8.821			8.834	8.826	
•	D4		8.852	8.842			8.836	8.840	
P	DI	Jr VII	8.822	8.822	8.821	8.827	8.831	8.797	
	D2	FM 5228-Rosette	8.816	8.840			8.819	8.811	
	D3 ·	+ Intremold III	. 8.826	8.810			8.806	8.815	
	D4	Ortho	8.839	8.838			8.825	8.944	
G	DI .	FM 5228	9.153	9.148	9.148	9.154	9.119	9.124	
	D2	Rosette	9.193	9.168			9.172	9.177	
	D3		9.155	9.127			9.103	9.143	
	D4		9.233	9.206			9.181	9.165	

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TABLE XI

CHANGE IN OUTSIDE DIAMETER OF AGCARD LINER/INTREMOLD III PIN 2002

			Changes in Outside Diameters between Process Steps							
Outside : Loc. Section	Diameter ation Position	Material	AO.D. Graph. to 2nd Imp. Carb.	AO.D. 2nd to 3rd Imp. Carb.	ΔO.D. 3rd to 4th Imp. Carb.	40.D. 1st Graph. to 4th Imp. Carb.	40.D. 4th Imp. Carb. to Regraph.	AO.D. lst Graph. to Regraph.	AO.D. Orig. Cure . to	
Section	rosition	naterial	Care.	Carb.	caro.	Car 5.	Kegtapa.	Kextapa.	Regraph.	
A	D1 D2 D3 D4	FM 5228 Rosette	039	+,010	039	063	+.036 039 +.077 025	027 054 005 038	045 088 035 075	
В	D1 D2 D3 D4	JTXVI FM 5228 Rosette Intremold III Ortho	003	+.006	+.003	+.006	+.029 002 +.019 +.909	+.035 015 +.001 +.034	+.103 024 011 004	
·c	D1 D2 D3 D4	Intremold III Ortho	+.039	+.008	063	016	008 010 003 001	024 005 015 005	020 029 011 017	
D	D1 D2 D3 D4	Intremold III Ortho	+,001	÷.006	024	017	+.005 +.002 +.011 +.004	013 006 +.003 +.002	017 023 607 014	
ε.	D1 D2 D3 D4	Intremold III Ortho	002	+.003	027	021	008 +.029 008 +.004	029 +.005 +.005 002	028 +.013 012 012	
r	D1 D2 D3 D4	JT VII FM 5228-Rosette + Intremold III Ortho	001	+.006	+.004	+.009	034 008 009 +.019	025 029 +.005 +.006	025 005 011 +.005	
G	D1 D2 D3 D4	FM 5228 Rosette	.000	+.005	035	029	+.055 +.005 +.040 016	024 +.009 +.016 041	029 016 012 068	

PERCEPT CHANGE IN OUTSIDE DIAMETERS OF AGGARD LINER/INTREMOLD III
CYLINDER 2001

			Percentage A O.D. Between Process Steps									
	Diameter ation Position	Material	<pre>% A 0.D. Graph to 2nd Imp. Carb.</pre>	% A C.D. 2nd to 3rd Imp. Carb.	% A O.D. 3rd to 4th Imp. Cerb.	% A C.D. lst Graph to lth Imp. Carb.	X A O.D. hth Imp. Carb. td Regraph	% A O.D. Let Graph to Regraph	% 4 0.D. Orig. Cure to Regreph			
			00101	OHLES	00.152	0.10	110770014	rogradit.	HOP, I C. P. I			
A	D1 D2 D3 D4	Intremold III Helix	+•01	+.01	06	03	+.1:3 +.24 +.50 06	+.36 +.23 +.11 +.14	+.60 +.33 +.36 +.39			
	•					Avg.	+.24	+.21	+.42.			
3	D1. D2 D3 Dh	FM 5228 Shingle	Olı	08	10	22	+.19 +.02 +.04 +.26	03 +.11 +.04 +.15	17 09 13 +.0h			
				•		Avg.	+.13	+.07	09			
C	D1 D2 D3 D4	Intremold III Helix	+• 03	+.01	+•07	+•11	+.38 +.27 +.16 +.83	+.50 +.76 +.11 +.41	+.26 +.50 +.11 +.28			
	•			•		Avg.	+.41	+.45	+.36			
ע	D1 D2 D3 D4	Joint II Intremold III FM 5228 Shingle	-•02	+•Ch	02	-•n	+.20 +.22 +.08 +.51	+.09 +.03 03 +.27	+.03 +.01 +.03 +.35			
						Avg.	*.25	+.12	+.11			
Z	D1 D2 D3 D14	FM 5228 Shingle	10	04	+•02	12	+.06 +.33 1h	05 +.20 28	29 05 1:0			
	Ъц					Ave •	+.12	+.38 +.16	+ <u>.05</u>			

TABLE XIII

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCaib Liner/Intremold III CYLINDER PIN 2002

•			Percentage AO.D. between Process Steps							
Outside i	ation		7 AO.D. Graph. to 2nd Imp.	% AO.D. 2nd to 3rd Imp.	% AO.D. 3rd to 4th Imp.	% AO.D. lst Graph. to 4th Imp.	7 AO.D. 4th Imp. Carb. to	Z AO.D. 1st Graph. to	% AO.D. Orig. Cure to	
Section	Position	<u>Material</u>	Carb.	Carb.	Carb.	Carb.	Regraph.	Regraph.	Regraph.	
A	D1 D2 D3 D4	FM 5228 Rosette	37	+.11	43	69 · Avg.	+.39 43 +.30 <u>27</u> 03	29 59 05 <u>42</u> 34	49 96 38 82 65	
В	D1 D2 D3 D4	JTXVI FM 5228 Rosette Intremold III Ortho	-1.03	+.05	03	+.07 Avg.	+.32 02 +.21 +.10 +.15	+.39 17 +.01 +.36 15	+1.16 ⁽¹⁾ 2712 <u>04</u> +.21	
С	D1 D2 D3 D4	Intremold III Ortho	+.44	+.09	71	18 Avg.	09 11 03 01 06	03 06 17 06 14	23 33 12 19 12	
D .	D1 D2 D3 D4	Intremold III Ortho	+.01	+.07	27	19 Avg.	+.06 +.02 +.12 +.05 +.06	15 07 +.03 +.02 04	19 26 08 16 17	
8	D1 D2 D3 D4	Intremold III Ortho	02	+.09	31	24 Avg.	09 +.33 09 +.05 +.05	33 +.06 +.09 02 05	32 +.15 14 14 03	
7	D1 D2 D3 D4	JT VII FM 5228-Rosette + Intremold III Ortho	01	+.07	+.05	+.10 Avg.	39 04 10 +.22 09	28 33 +.06 +.07 12	39 06 12 +.06 13	
G .	D1 D2 D3 D4	FM 5228 Rosette	.000	+.07	38	- .35 Avg.	+.06 +.06 +.44 17 +.10	26 +.10 +.18 <u>45</u> 11	32 17 13 74 34	

⁽¹⁾ Questionable number - average not meaningful

TABLE XIV

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGGARD LINER/INTREMOLD III CYLINDER PIN 2003

	•		Percentage & O.D. Between Process Steps									
Outside Loca Section		Material	% 40.D. Graph. to 2nd Imp. Carb.	% AO.D. 2nd to 3rd Imp Carb.	% AO.T. 2nd to hth Irp. Carb.	% AO.D. lst Graph. to lth Imp. Carb.	% 40.D. hth Imp. Carb. to Regraph.	% 40.D. 1st Graph. to Regraph.	% AO.D. Orig. Cure to Regraph.			
A	D1 D2 D3 D4	FM 5228 Rosette	-•11	-	17 Avg.	28 13 0h 28 21	+.19 01 +.17 +.31 +.17	09 1h +.14 +.02 02	31 11 09 23 27			
B .	D1 D2 D3 D4	JT-V Intremold III Helix FM 5228 Rosetto	-•02	-	13 Avg.	66 +.13 11 14 27	+.77 +.1.1 +.55 +.72 +.61	+.10 +.54 +.43 +.27 +.34	+.61 +.45 +.54 +.55			
c	ph p3 p1	Intremold III Helix	0.00		-•11 Avg.	11 +.1:8 +.25 +.01 +.16	+.68 +.17 +.27 +.39 +.38	+.57 +.65 +.52 +.1:0 +.54	• .92 •1.02 • .86 • .63 • .86			
D	D1 D2 D3 D4	Intremold III Helix	+•03	-	49 Avg.	45 +.11 +.01 15 12	+.90 +.78 +.1,1 +1.07 +.80	+•lili +•98 +•li5 +•92 +•60	+.17 +.67 +.70 +.64 +.55			
E	D1 D2 D3 D1,	Intremold III Helix	 02	. -	60 Avg.	62 +.01 23 14 25	+.12 +.26 +.24 +.32 +.31	20 +.27 +.01 +.18 +.07	+.12 0.00 +.05 +.18 +.09			
P	DH D3 D5 D1	JT VIII Intremold III Helix FM 5228 Rosette	07	•	llı Avg.	21 +.19 28 +.23 02	05 36 1:0 29 28	25 17 68 06 29	57 70 98 81 77			
G	DI D3 D1	FN 5228 Rosette	03	•	18 Avg.	22 +.30 07 +.21 +.06	+.20 +.16 10 +.23 +.12	01 +.46 17 +.43 +.18	06 +.29 21 +.21 +.06			

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PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCARD LINER/INTREMOLD III CYLINDER PIN 2004

		Percentage 4 0.D. Between Process Steps									
Outside Toca	Diameter tion	·	SAO.D. Graph. to 2nd Imp.	% A O.D. 2nd to 3rd Imp.	% 40.D. 3rd to 4th Imp.	t	540.D. St Graph. Ce	\$ 40.D. Uth Imp. Carb. to	% A O.D. lst Graph. to	% 40.D. Orig. Cure to	
Section	Position	Material	Carb.	Carb.	Carb.		arb.	Regraph.	Regraph.	Regraph.	
A	D1 D2 D3 D4	JT XVIII FM 5228 Rosette Intremold III Ortho	+•58 •	63	*• 03		+.0l ₁ +.05 +.16 17 +.02	66 115 117 58 514	62 40 32 75 52	91 83 70 -1.09 88	
В	סן 23 25 20	JT XVIII FM 5228 - Rosette Intremold III Ortho	07	+•01	21		27 11 +.0h 23 1h	08 39 27 22 24	36 19 23 15 38	48 62 18 66	
c .	ըր ը3 ը5	Intremold III Ortho	+•11	0.00	03		+.08 +.02 07 +.08 +.03	30 +-35 +-35 21 +-05	22 +.37 +.28 13 +.08	28 +.17 +.15 25 05	
D	סף 23 25 סד	Intremold III Ortho	+•03	~.20	+•30		+.17 +.07 +.08 +.28 +.15	13 +.25 02 38 07	+.03 +.31 +.06 10 +.08	09 +.19 06 30	
E .	D1 D2 D3 D4	JT XIX FM 5228-Rosette Intremold III Ortho	+• C9	17	+. 12		+.03 15 +.02 +.30 +.05	49 +.11 +.04 33 19	45 04 02 03 14	69 012 26 6h l:3	
?	D1 D2 D3 D4	FM 5228 Rosette	֥ 22	-•45	 c5		28 21; +.11; +.09 07	56 37 75 2h 18	84 61 62 11 55	83 87 88 30 72	

TABLE XVI

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCARD LINER/INTREMOLD III CYLINDER PIN 2005

			Percentage C.D. Between Process Steps								
Outside Loca Section		<u> </u>	% A O.D. Graph to 2nd Imp. Carb.	\$ a O.D. 2nd to 3rd Imp. Carb.	# AO.D. 3rd to lith Imp. Carb.	\$ 40.D. Lat Graph. to th Imp. Carb.	\$40.0. hth Imp. Carb. to Regraph.	% A O.D. 1st Graph. to Regraph.	% A O.D. Orig. Cire to Rogruph.		
.	D1 D2 D3 D4	PM 5228 Rosetta	 35	+•37	18 Avg	16 09 25 09 15	+.10 +.20 +.h5 +.02 +.19	05 +.11 +.20 07 +.05	+.15 12 +.29 03 +.07		
2	D1 D2 D3 DL	JT XX-FM 5228 Rosette Intremold III Helix	18	+.143	2lı Avg	0.00 +.06 07 09 03	16 09 +.16 19 07	16 03 +.09 29 010	15 27 02 39		
c .	D1 D2 D3 D4	Intremold III Helix	+.03	02	0.00 Avg	+.01 +.09 +.13 +.02 +.06	+.12 +.13 +.48 +.08 +.20	+.13 +.22 +.62 +.10 +.27	+.39 +.04 +.53 +.12 +.27		
. و	D1 D2 D3 D4	Intremold III Helix	*• 03	֥05	+.ll Avg	+.13 +.32 +.13 +.14 +.18	+.16 +.51 +.27 +.12 +.27	+.30 +.83 +.11 +.26 +.15	+.62 +.li9 +.39 +.32 +.li6		
E	D1 D2 D3 D4	JT XXIII FM 5228 Rosette Intremold III Helix	02	01	+•05 Avg	+.02 +.02 11 +.11 +.01	05 03 27 21	03 01 +.16 09 +.01	+.20 18 +.31 06 +.07		
F	D1 D2 D3 D4	FM 5228 Rosette	- <u>-</u> 0l4	-•06	02 Avg	12 +.01 04 +.02	+.08 10 +.06 25 05	0h 09 +.02 23 09	+.03 41 04 42 21		

TABLE XVIV

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2006

		Percentage A O.D. Between Process Steps								
Outside Loca Section	Diameter tion Position	Material	\$ 40.D. Graph. to 2nd Imp. Carb.	% AO.D. 2nd to 3rd Imp. Carb.	% AO.F. 3rd to lith Imp Carb.		% AO.D. 1st Graph. to 4th Imp. Carb.	% AC.D. hth Imp. Carb. to Rograph.	% AO.D. 1st Graph. to Regraph.	% AO.D. Orig. Cure to Rograph.
.	n D3 D4	JT IM FM 5228 Rosetto Intremold III Ortho	÷ <u>-</u> 06	20	+. 03	Avg.	11 05 11 07	55 +.34 21 24	65 +.28 32 32	69 01 55 18 13
В .	D1 D2 D3 D4	JT IM FM 5228 Rosette Intremold III Ortho	15	12	+.18	Avg.	08 +.02 08 0h	50 +.14 0h 17 1h	59 +.16 13 21 19	1:6 •.03 18 3!: 24
c	D1 D2 D3 D4	Intremold III Ortho	-•Ois	26	31	Avg.	61 03 33 05 26	+.1.5 +.61 +.1.9 +.1.1. +.1.2	16 +.57 +.15 +.09 +.16	21 +-33 +-04 12 +-01
Þ .	D1 D2 D3 D14	Intremold III Ortho	-•07	 05	03	A v g.	13 02 +.01 08 06	25 •.13 +.28 +.11 +.16	38 +.16 +.29 +.07 +.11	3h +-23 08 05
K	D1 D2 D3 D4	JT XVI FM 5228 Rosette Intremold III Ortho	-•07	-1.33	+1.34	A v g.	08 05 11 08 08	13 00 01 05 12	51 05 12 13 20	28 14 11 16 17
F	D1 D2 D3 D1,	JT XVI FM 5228 Rosette Intremold III Ortho	03	01	 08	Avg.	12 -1.11 05 -1.18	46 +1.04 -1.27 +1.06 +.09	57 08 24 14	35 12 22 16 21

TABLE X/III

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCATA LINER/INTREMOLD III CYLINDER PIN 2001

			Percentage AI.D. between Process Steps						
Inside D: Loca Section	iameter ation Position	<u>Material</u>	ZAI.D. Graph. to 2nd Imp. Carb.	ZAI.D. 2nd to 3rd Imp. Carb.	ZAI.D. 2nd to 4th Imp. Carb.	ZAI.D. 1st Graph. to 4th Imp. Carb.	ZAI.D. 4th Imp. Carb. to Regraph.	ZAI.D. lst Graph. to <u>Regraph.</u>	7AI.D. Orig. Cure to Regraph
· A	D1 D2 D3	Intremold III Helix	29	 	13	40 Avg.	+.51 +.30 +.57 +.46	+.09 01 +.09 +.06	+.93 +.79 +.79 +.84
В	D1 D2 D3	FM 5228 Shingle	+.03		13	10 Avg.	+.11 +.15 +.28 +.18	+.01 · +.03 · +.05 · +.03	+.36 +.33 +.41 +.37
, c	D1 D2 D3	Intremold III Helix	03	 	23	25	+.52 +.50 +.51	+.27 +.36 +.25 +.29	+.55 +.59 +.78 +.64
D	D1 D2 D3	Joint II Intremold III Helix FM 5228 Shingle	06		28	34 Avg.	+.65 +.65 +.56 +.61	+.30 +.19 +.20 +.23	+.59 +.51 +.46 +.52
Z .	D1 D2 D3	FM 5228 Shingle	09		14	22 Avg.	+.47 +.36 +.34 +.39	+.24 +.18 +.10 +.17	+.52 +.40 +.37 +.43

TABLE XIX

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCARD LINER/INTREMOLD III CYLINDER PIN 2002

•			Percentage ΔI.D. between Process Steps								
Inside D			7Δ1.D. Graph. to	7.67.D. 2nd to	7AI.D. 2nd to	7AI.D. lst Graph. to	751.D. 4th Imp. Carb.	7Δ1.D. lst Graph.	%MI.D. Orig. Oure		
	ation		2nd Imp.	3rd Imp.	4th Imp.	4th Imp.	to	to	, to		
Section	Position	<u>Material</u>	Carb.	Car-b.	Carb.	Carb.	Regraph.	Regraph.	Regraph.		
A	Dl	FM 5228	29		+.04	25	4.08	18	+.15		
	D2	Rosette				+.27	+.05	22	+.05		
	D3				•	29 27	<u>†.11</u>	<u>18</u>	<u>01</u>		
					Avg.	27	+.08	19 .	+.06		
В	DI	JTXVI - FM 5228	23		20	43	1.32	11	+.18		
	D2	Rosette - Intremold				34	4.15	19	+.15		
	D3	III Ortho				44 40	<u> </u>	19	. +.24		
					Avg.	40	24	19 16	+.24 +.19		
c	Dl	Intremold III			05	28	ء.29	+.01	+.19		
	D2	Ortho		••		34	1.23	06	+.23		
	D3					<u>29</u>	+.24	05	05 %		
					Avg.	<u>29</u> 30	+.27	03	+.23 05 +.12		
D	Dl	Intremold III	19		10	29	+.20	09	+.23		
	D2	Ortho				24	+.18	09	+.23		
	D3			~~		28 27	+.20	08 09	<u>+.31</u>		
	•				Avg.	27	+.19	09	+.26		
E	Dl	Intremold III	16		20	37	+.45	+.08	+.28		
	D2	Ortho				33	+.38	+.05	429		
	D3					<u>44</u>	+.47	<u>+.03</u>	<u>+.28</u>		
		,			Avg.	38	+.43	+.05	+.28		
P	Dl	JTVII - FM 5228	20		15	36	+.29	06	+.14		
	D2	Rosette - Intremold				38	+.29	09	+.11		
	D3	III Ortho				<u>41</u>	<u>+.25</u>	15 10	+.15		
					Avg.	41 38	+.28	10	+.15 +.13		
. G	D1	FM 5228	22		09	30	+.15	15	+.14		
	D2	Rosette				33	+.18	15	+.13		
	D3					<u>36</u>	+.20	<u>15</u>	<u>+.18</u>		
					Avg.	33	+.18	15	+.15		

TABLE XX

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCARD LINER/INTREMOLD III CYLINDER PIN 2003

•			Percentage ΔI.D. between Process Steps									
			741.D.	7ΔI.D.	%ΔI.D.	7ΔI.D.	7A1.D.	%ΔI.D.	ZAI.D.			
			Craph.	2nd	2nd	lst Graph.	4th Imp.	lst	Orig.			
Inside Di			to	to	to	to	Carb.	Graph.	Cure			
Loca	ation		2nd Imp.	3rd Imp.	4th Imp.	4th Imp.	to	to	. to			
Section	Position	Material	Carb.	Carb.	Carb.	Carb.	Regraph.	Regraph.	Regraph.			
A	D1	FM 5228	04		43	47	÷.50	+.03	+.19			
	D2	Rosette				48	+.53	+.05	+.29			
	D3			**		44 46	+.48 +.50	±.04 +.04	<u>+.28</u> +.25			
					Avg	46	+.50	+.04	+.25			
В	Dl	JTV Intremold	01		28	29	+.31	+.01	+.32			
	D2	III Helix				34	+.38	+.04	+.34			
	D3	FM 5228 - Rosette				34 32	+.46 +.38	+.11 +.05	+.43 +.36			
					Avg	32	+.38	+.05	+.36			
С	Dl	Intremold III	03		39	42	+.38	+.01	+.42			
	D2	Helix				28	+.28	.00	+.56			
	D3					34 35	+.39 +.35	+.05	+.56 +.52 +.50			
					Avg	35	+.35	+.02	+.50			
D	Dl	Intremold III	01		23	24	+.04	20	÷.34			
	D2	Helix				24	+.04	20	+.32			
	D3					30 26	+.14	16 19	+.34 +.33			
					Avg	26	+.07	19	+.33			
, E	D1	Intremold III	04	••	18	22	+.10	11	+.34			
	D2	Helix				25	+.14	11	+.36			
•	D3		•			34 27	$\frac{+.22}{+.15}$	14 12	+.39 +.35			
					Λvg	27	+.15	12	+.35			
P	D1 .	JTXVIII Intremold	05		24	29	+.50	+.20	+.51			
	D2	III Helix - FM 5228				34	÷.48	+.14	+.41			
	D3	Rosette				25 29	+.42 +.47	+.16 +.17	+.53			
					Avg	29	+.47	+.17	+.50			
G	D1	FM 5228	06		15	34	+.32	02	+.41			
	D2	Rosette				39	+.39	.00	+.48			
	D3				•	33 35	<u>+.31</u>	03 02	+.43			
					Avg	35	+.34	02	+.44			

TABLE XXI

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCORD LINER/INTREMOLD III CYLINDER PIN 2004

					Percent	age AI.D	. between Proce	ss Steps		
			7ΔI.D. Graph.	7ΔI.D. 2nd	741.D. 3rd		MAI.D. 1st Graph.	%AI.D. 4th Imp.	7ΔI.D. Ist	ZAI.D. Orig.
Inside Di			to	to	to		to	Carb.	Graph.	. Cure
	ation		2nd Imp.	3rd Imp.	4th Imp	·.	4th Imp.	to	to	to
Section	Position	<u>Material</u>	Carb.	Carb.	Carb.	_	Carb.	Regraph.	Regraph.	Regraph.
A	D1	JTXVIII - FM 5228	09	09	+.01		17	+.04	13	14
	D2	Rosette - Intremold					09	08	17	14
	D3	III Ortho					10 12	+.09	01 10	03 10
						Avg.	12	+.02	10	10
В	D1	JTXVIII - FM 5228	18	+.06	14		15	+.14	01	01
	D2	Rosette - Intremold					15	+.18	+.03	05
	D3	III Ortho					19 16	+.28	+.09	+.03 04
						Avg.	16	+.20	+.04	04
С	D1	Intremold III	56	04	+.05		54	+.28	27	+.10
	D2	Ortho					52	+.43	09	+.23
	D3						54 53	+.45 +.39	<u>10</u>	+.25 +.19
						Avg.	53	+.39	15	+.19
D	Dl	Intremold III	52	03	+.03		52	+.31	22	+.09
	D2	Ortho					49	+.37	13	+.18
	D3						52 51	+.43 +.37	09 15	+.22 +.15
						Avg.	51	+.37	15	+.15
E	ָ בו	JTXVIII - FM 5228	38	07	+.03	•	43	+.20	23	08
	D2	Rosette - Intremold					46	+.19	27	09
	D3	III Ortho					43 44	+.22 +.20	22 24	<u>06</u>
•						Avg.	44	+.20	24	08
7	Dl	FM 5228	14	+.07	10		17	+.11	05	+.01
	D2	Rosette					11	+.09	03	+.03
	D3						14 14	+.15 +.12	+.01	
						Avg.	14	+.12	03	+.05 +.03

TABLE JXII

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCARD LINER/INTREMOLD III CYLINDER PIN 2005

			Fercentage ALD, between Process Steps								
			7AI.D.	7ΔI.D.	%∆I.D.	741.D.	7AI.D.	7AI.D.	ZAI.D.		
			Graph.	2nd	3rd	lst Graph.	4th Imp.	lst	Orig.		
Inside D			to	to	to	to	Carb.	Graph.	Cure		
-	ation	**	2nd Imp.	4th Imo.	4th Imp.	4th Imp.	to	to	to		
Section	Position	Material	Carb.	Carb.	Carb.	Carb.	Regraph.	Regraph.	Regraph.		
A	D1	FM 5228	28	05	07	40	+.28	12	+.27		
	D2	Rosette				37	+.80	+.43	+.65		
	D3					<u>28</u> 35	+.33 +.47	+.05 +.12	+.22 +.38		
					Avg.	35	+.47	÷.12	+.38		
В	D1	JTXX - FM 5228	26	+.21	16	21	+.20	01	+.12		
	D2	Rosette - Intremold				41	+.63	÷.27	+.42		
	D3	III Helix				4 <u>1</u>	+.41 +.43	0.00 +.09	$\frac{+.13}{+.22}$		
					Avg.	34	+.43	+.09	+.22		
С	DI	Intremold III	+.17	15	07	05	+.37	+.32	+.48		
	D2	Helix				- .37	+.70	+.33	+.50		
	D3					- .34		12			
					Avg.	34 25	+.22 +.43	12 +.18	02 +.32		
D	D1	Intremold III	07	27	+.17	17	+.38	÷.21	+.49		
	D2	Helix				18	+.44	426	+.49		
	D3					21	+.25	+.04	+.28		
					Avg.	21 19	+.36	+.04 +.17	+.42		
Ε.	Dl	JTXXIII - FM 5228	38	+.09	+.06	23	+.30	+.06	+.39		
	D2	Rosette - Intremold				32	+.49	+.17	+.37		
	D3	III Helix				42	+.25	17			
					Avg.	42 32	+.35	17 +.02	+.10 +.29		
F	D1	FM 5228	42	+.20	11	33	+.27	 C6	+.25		
	D2	Rosette		· · · ·	5 557	42	+.65	+.23	+.46		
	D3						<u>17</u>		+.07		
					Avg.	31 35	+.36	14 +.01	+.26		

TABLE XXIII

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCAPD LINER/INTREMOLD III CYLINDER PIN 2006

					Percentage AI	.D. between Proce	ess Steps	1		
Inside Di Loca Section	iameter stion Position	<u> Material</u>	7AI.D. Graph. to 2nd Imp. Carb.	ZAI.D. 2nd to 3rd Ymp. Carb.	ZAI.D. 3rd to 4th Imp. Carb.	7AI.D. 1st Graph. to 4th Imp. Carb.	%AI.D. 4th Imp. Carb. to Regraph	7Δ1.D. lst Graph. to Regraph.	751.D. Orig. Cure to Regraph.	
A	D1 D2 D3	JT IM FM 5228 - Shingle Intremold III Ortho	15	08	.00	23 11 18 17	+.34 +.27 +.22 +.27	+.11 +.15 +.04 +.10	+.38 +.38 +.25 +.34	
В	D1 D2 D3	JT IM FM 5228 - Rosette Intremold III Ortho	34	03	01 Avg.	38 43 37 39	+.24 +.30 <u>+.24</u> +.26	14 13 13 13	+.10 +.05 +.05	
c	D1 D2 D3	Intremold III Ortho	32	15	+.05 Avg.	42 25 42 36	+.33 +.22 <u>+.29</u> +.28	09 04 13 09	+.09 +.08 04 +.03	いたが
D .	D1 D2 D3	Intremold III Ortho	44	04	+.11 Avg.	37 40 44 40	+.33 +.37 <u>+.37</u> +.36	04 04 03 05	+.09 +.03 +.03 +.05	
	D1 D2 D3	JTXVI - FM 5228 Rosette Intremold III Ortho				 	 A∀g.	13 19 15 16	+.13 06 +.05 +.04	
P	D1 D2 D3	JTXVI - FM 5228 Rosette Intremold III Ortho	20	09	.00 Avg.	29 29 22 27	+.32 +.27 +.11 +.23	+.03 03 10 03	+.38 +.36 +.34 +.36	

TABLE XXIV

CHANGE IN LENGTH OF AGGARD LINER/INTREWOLD III CYLINDERS 2001, 2002, 2003, 2004, 2005 & 2006

		Position	•		s Step Length ts in Inches			% A L lst Oraph	% A L Ltk Imp. Carb.	% & L Graph	% & L Orig.	
Cylinder Number	Material	of Length Measure- ment	Orig.	First Graph.	hth Imp. Carb.	Regraph.		to 4th Imp. Carb.	to Regraph.	%o Regraph.	to Regraph.	
2001	FM 5228 Shingle/ Intremold III Helix	0° 90° 180° 270°	17.940 17.942 17.955 17.945	17.891 17.900 17.890 17.900	17.852 17.864 17.871 17.870	17.893 17.900 17.890 17.897	Avg.	22 20 11 17 18	+.17 +.20 +.11 +.15 +.15	05 .00 .00 02	31 23 36 27 29	
2002	FM 5228 Rosette/ Intremold III Orthogonal	0° 90° 180° 270°	16.031 16.031 16.000 16.039	15.997 16.012 16.019 16.033	15.964 15.983 16.008 15.998	15.982 15.998 16.002 16.002	A∀g•	21 18 07 22 17	+.E1 +.09 09 +.03 +.05	09 09 11 19 12	31 21 +-01 23 18	(
2003	FM 5228 Rosette/ Intremold III Helix	0° 90° 180° 270°	16.569 16.649 16.646 16.614	:	16.590 16.619 16.569 16.546	16.630 15.564 16.612 16.540	Avg.	- - -	+.24 33 +.26 +.57 +.19	-	+.37 51 20 +.16 05	
20011	FM 5228 Rosette/ Intremold III Orthogonal	0° 90° 180° 270°	15.979 16.012 15.996 15.968	15.985 16.032 15.985 15.982	15.962 15.980 16.000 15.964	15.970 15.981 15.977 15.982	Avg.	14 32 09 11 17	+.05 +.01 14 +.11 +.01	09 32 05 0.00 12	06 19 12 +.09 07	
2005	FM 5228 Rosette/ Intremoid III Helix	0° 90° 180° 270°	17.416 17.403 17.391 17.394	17.298 17.285 17.285 17.284	17.293 17.260 17.261 17.263	17.253 17.234 17.240 17.248	A∀g.	03 14 12 11	23 15 12 09 15	26 30 26 21 26	94 97 92 84 92	
2006	FM 5228 Rosette and Shingle/ Intremold III Orthogonal	0° 90° 160° 270°	14.585 14.580 14.638 14.593	14,693 14,580 14,610 14,513	14,556 14.544 14.605 14.517	14.585 14.559 14.627 14.517	Avg.	0.32 25 03 +.03	+.20 +.10 +.15 00 +.11	12 14 +.11 +.03	co lh c8 52	

TAFLE XXV

RESULTS OF ISOPROPYL ALCOHOL PERMEABILITY⁽¹⁾ AND FENETRANT TESTS ON AGCARD LINER/INTREMOLD III CYLINDERS PIN 2001, 2002, 2003, 2004, 2005 AND 2006

Cylinder Number	<u>Material</u>	Circumferential Location of Alcohol Test	Axial Location of Alcohol Test	Time to Fenetrate Wall I.D. to O.D. After 4th Imp. Carb., seconds	Time to Penetrate Wall I.D. to O.D. After Regraph., seconds	Alcohol Penetrant ⁽²⁾ Results After Regraph.
2001	Intremold III Eelix	0° 90° 180° 270°	Center of Intremold III Area C Section	 	11.5	Numerous intermittant delans. in O.D. roving. No other alcohol indications. Intremold sections porous.
	FM 5228 Shingle	0° 180°	A Section		No No	
	Roving Area	0° 180°	D Section	Ξ.	No No	3
2002	Intremold III Orthogonal	0° 90° 180° 270°	D Section	 	9.0 8.5 8.3 10.0	No indications of delaminations or separations; however, Intremold section was porous.
	FM 5228 Rosette	0° 180 ⁹	A-B Section	 	No No	
2003'	Intremold III Relix	0° 90° 180° 270°	D-E Section	 	22.0 15.5 18.1 8.9	No indications of delaminations or separations; however, variable porosity in Intremold area.
	FM 5228 Rosette	0° 180°	A-B Section		No No	<u>.</u>
2004	Intremold III Orthogonal	0° 90° 180° 270°	C-D Section	No No No	9.0 12.9 14.6 13.2	No indications of delaminations; however, Intremold sections were porous.
	FM 5228 Rosette	0° 90° 180° 270°	Both A & F Sections	No No No No	No No No No	

⁽¹⁾ The alcohol permeability test was accomplished by pouring 10 cc of isopropyl alcohol on the selected test area and visually checking the time to penetrate through the wall. If no penetration occurred, a (no) was recorded in the time column, otherwise the time for the first sign of wetness on the 0.D. was recorded as the time to penetrate.

⁽²⁾ Alcohol penetrant test for delaminations and/or separations was made in accordance with AGC Specification 36491.

TABLE XXV (cont.)

RESULTS OF ISOPROPYL ALCOHOL PERMEABILITY (1) AND PENETRAIT TESTS ON AGCARD LINER/INTREMOLD III CYLINDERS 2001, 2002, 2003, 2004, 2005 AND 2006

Cylinder Number	<u> Material</u>	Circumferential Location of Alcohol Test	Axial Location of Alcohol Test	Time to Penetrate Wail I.D. to O.D. After 4th Imp. Carb., seconds	Time to Penetrate Wall I.D. to O.D. After Regraph., seconds	Alcohol Penetrant ⁽²⁾ Results After Regraph.
2005	Intremold III	0° 90°	C-D Section	No No	8.0	No indications of delaminations
	Helix	180°		No No	7.9 6.1	or separations; however, Intremold sections were porous.
		270°		No	5.0	intremota sections were porous.
				•		
	FM 5228	0°	A Section	No	No	
	Rosette	900		No	No	Ć
•		180° 270°		No	Мо	•
		270°	•	No	No	,
2006	Intremold III	o°	C-D Section	No	15.1	No specific indications of
	Orthogonal	90 ⁰	• • • • • • • • • • • • • • • • • • • •	No	18.1	delaminations observed after
	Ü	180° 270°		No	30.0	final graphitization; however,
		270°		No	25.0	porosity was observed in AGCarb
						rosette at 270°. Note: The AGCarb
	FM 5228	o° a	A Section	No	No	IM shingle showed delaminations
	Shingle	900		No	No	for almost 360°, .050 to .130 from
		1800		No	No	I.D. after original graphitization.
		270 ⁰		No	No	

⁽¹⁾ The alcohol permeability test was accomplished by pouring 10 cc of isopropyl alcohol on the selected test area and visually checking the time to penetrate through the wall. If no penetration occurred, a (no) was recorded in the time column, otherwise the time for the first sign of wetness on the I.D. was recorded as the time to penetrate.

⁽²⁾ Alcohol penetrant test for delaminations and/or separations was made in accordance with AGC Specification 36491.

TABLE XXVI

MEANS AND STANDARD DEVIATIONS FOR MAJOR PROCESS PARAMETERS AND CYLINDER PROPERTIES

1. Cylinder Starting Specific Gravity

Sample size n = 6

Mean, $\overline{x} = 1.023$

Standard Deviation S.D. = .0408

2. Cylinder Final Specific Gravity

Sample size n = 6

Mean, $\overline{x} = 1.431$

Standard Deviation S.D. = .0215

3. Percent Weight Pick up First Impregnation Based on Starting Cylinder Weight

Sample size n = 6

Mean, $\bar{x} = 35.683$

Standard Deviation S.D. = 6.778

4. Cylinder Specific Gravity after First Impregnation Carbonization

Sample size n = 6

Mean, $\overline{x} = 1.267$

Standard Deviation S.D. = .030

5. Cylinder Specific Gravity after Second Impregnation Carbonization

Sample size n = 6

Mean, $\overline{x} = 1.347$

Standard Deviation S.D. = .022

6. Cylinder Specific Gravity after Third Impregnation Carbonization

Sample size n = 6

Mean, $\overline{x} = 1.396$

Standard Deviation S.D. .029

TABLE XXVI

7. Cylinder Specific Gravity after Fourth Impregnation Carbonization

Sample size n = 6

Mean, $\overline{x} = 1417$

Standard Deviation S.D. = .028

8. Percent Weight Pick up Second Impregnation Based on Cylinder Weight

Sample size n = 6

Mean $\overline{x} = 16.78$

Standard Deviation S.D. = 6.256

9. Percent Weight Pick up Third Impregnation Based on Cylinder Weight

Sample size n = 6

Mean $\overline{x} = 12.0$

10. Percent Weight Pick up Fourth Impregnation Based on Cylinder Weight

Sample size n = 6

Mean $\overline{x} = 11.133$

Standard Deviation S.D. = 2.016

11. 15V /Indene - Viscosity After Impregnation

Sample size n = 13

Mean $\overline{x} = 105.538$

12. 15V/Indene - Specific Gravity After Impregnation

Sample size n = 13

Mean $\overline{x} = 1.165$

Standard Deviation S.D. = .015

TABLE XXVI (cont)

13. 15V/Indene - Temperature After Impregnation

Sample size n = 13

Mean $\bar{x} = 251.385$

14. 15V/Indene - Temperature Before Impregnation (all impregnations)

Sample size n = 24

Mean $\overline{x} = 252.083$

Standard Deviation S.D = 14.981

15. 15V/Indene Percent Volatiles Based on Weight Loss During Cure (all impregnations)

Sample size n = 24

Mean $\bar{x} = 25.833$

Standard Deviation S.D. = 9.994

16. 15V/Indene Percent Carbon Yield Based on Cured Impregnation Weights

Sample size n = 24

Mean x = 57.358

Standard Deviation S.D. = 13.245

17. 15V/Indene Percent Pick up Based on Cylinder Weights (all impregnations)

Sample size n = 24

Mean $\overline{x} = 27.5$

18. 15V/Indene Percent Carbon Yield Based on Impregnation Weights (all impregnations)

Sample size n = 24

Mean $\frac{-}{x} = \frac{1}{42.1479}$

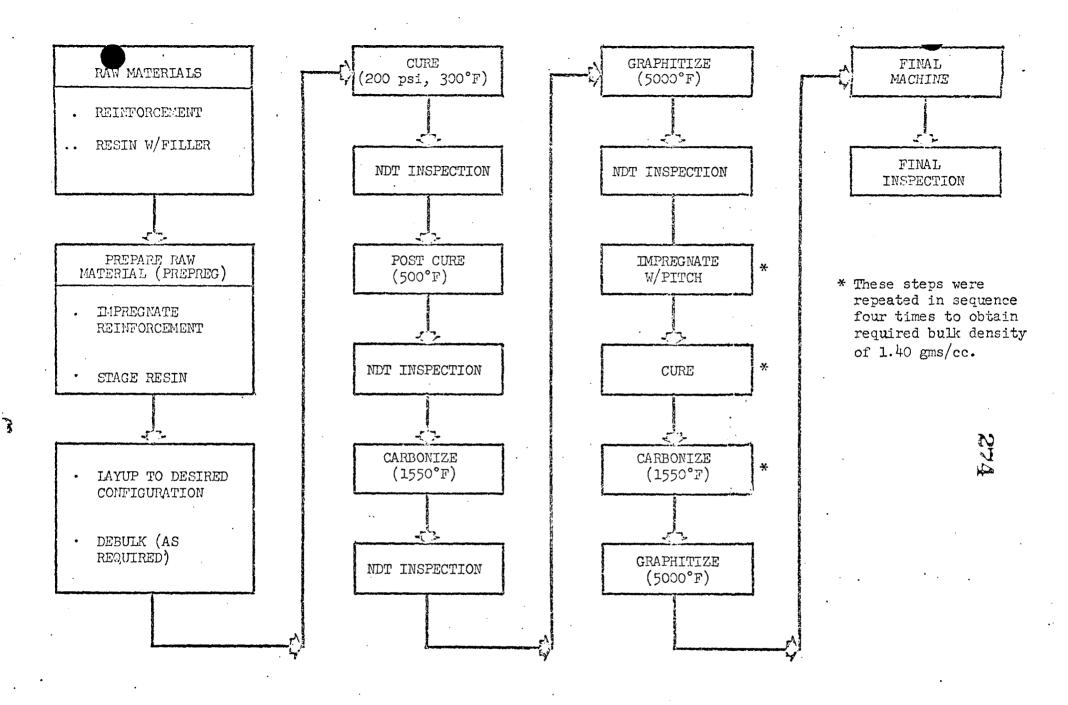


Figure 1. AGCarb Liner/Intremold III Cylinder, Fabrication Sequence

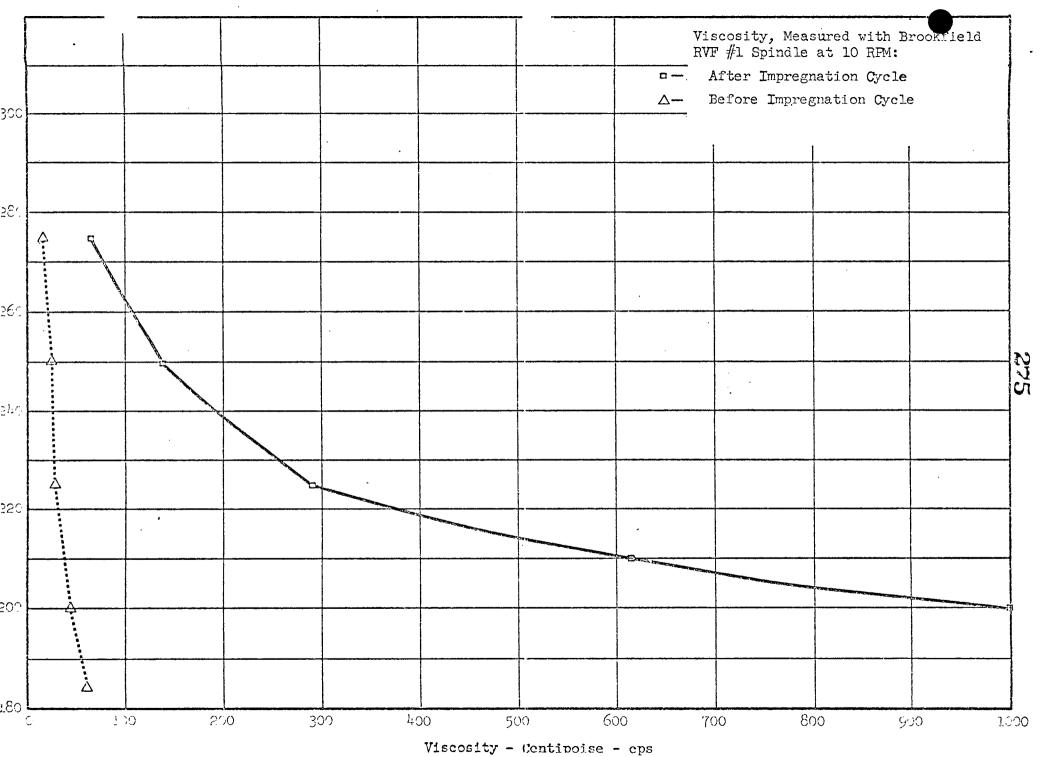


Figure 2. Effect of Temperature on Viscosity of 15V/Indene

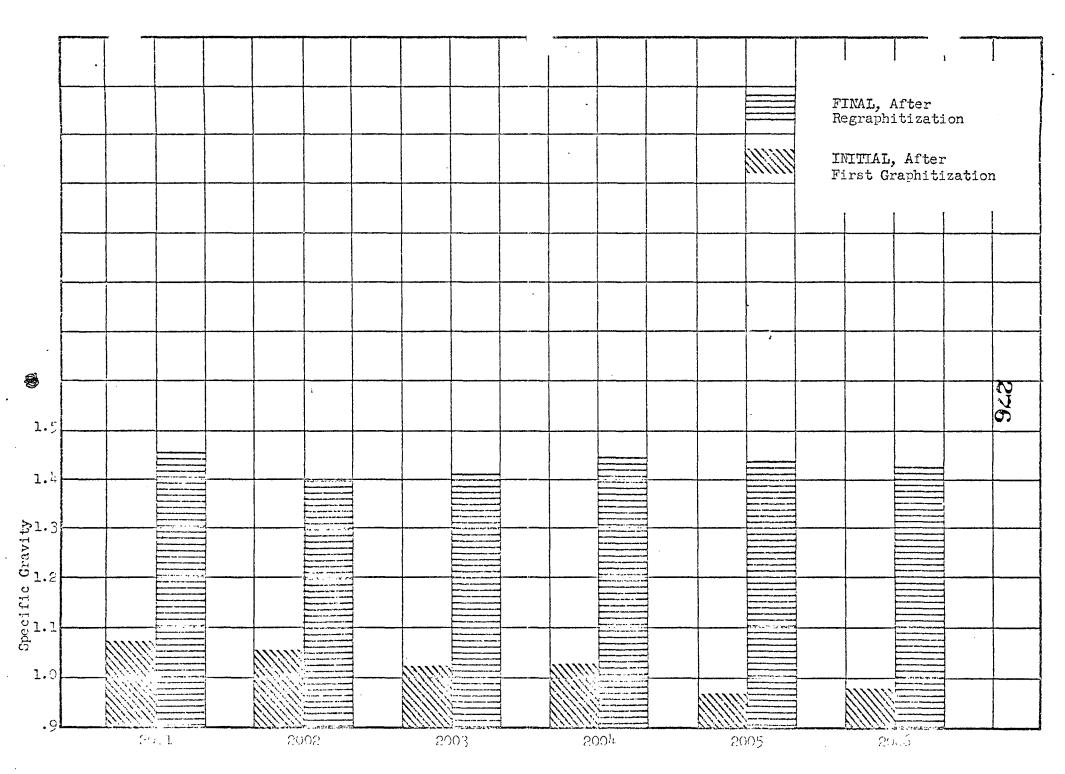


Figure 3. Specific Gravity of Cylinders Number 2001 Through 2006

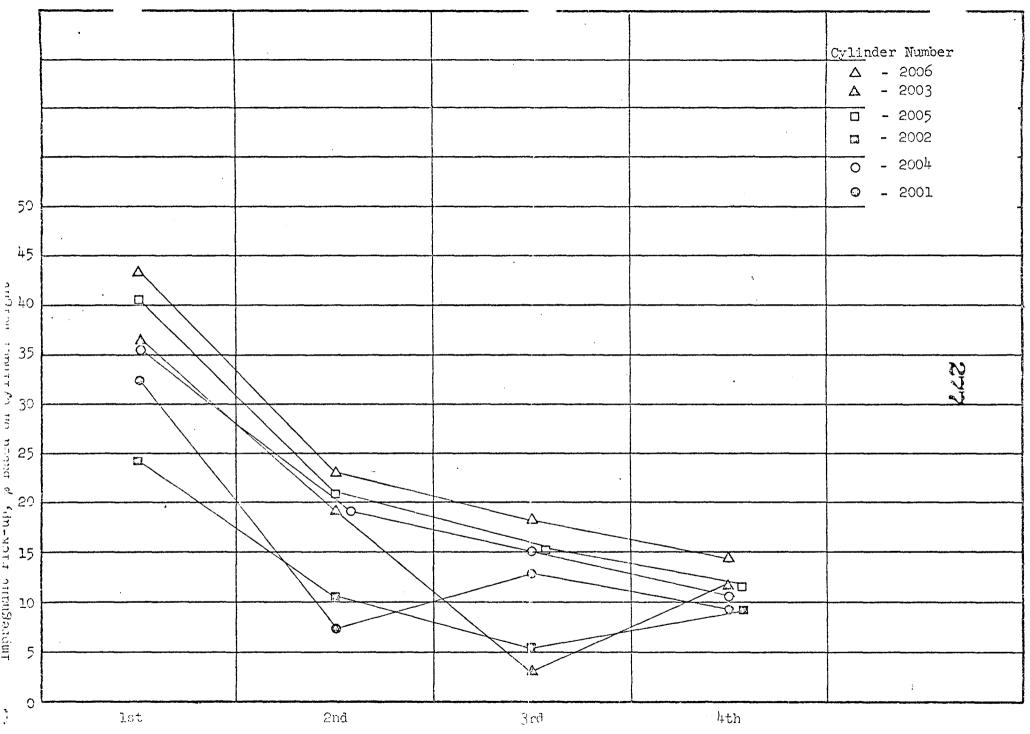


Figure 4. 15V/Indene Impregnant Pick-up Versus Impregnation Cycle

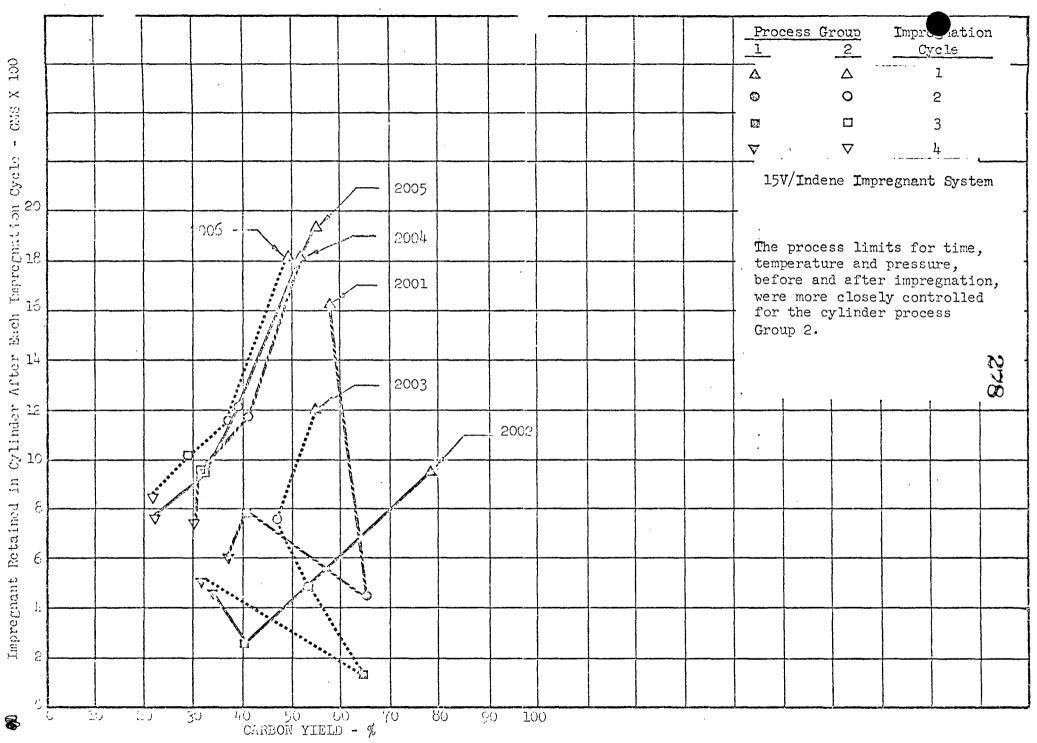


Figure 5. Effect of Process Control on Impregnant Retention and Carbon Yield in Cylinders 2001 through 2006

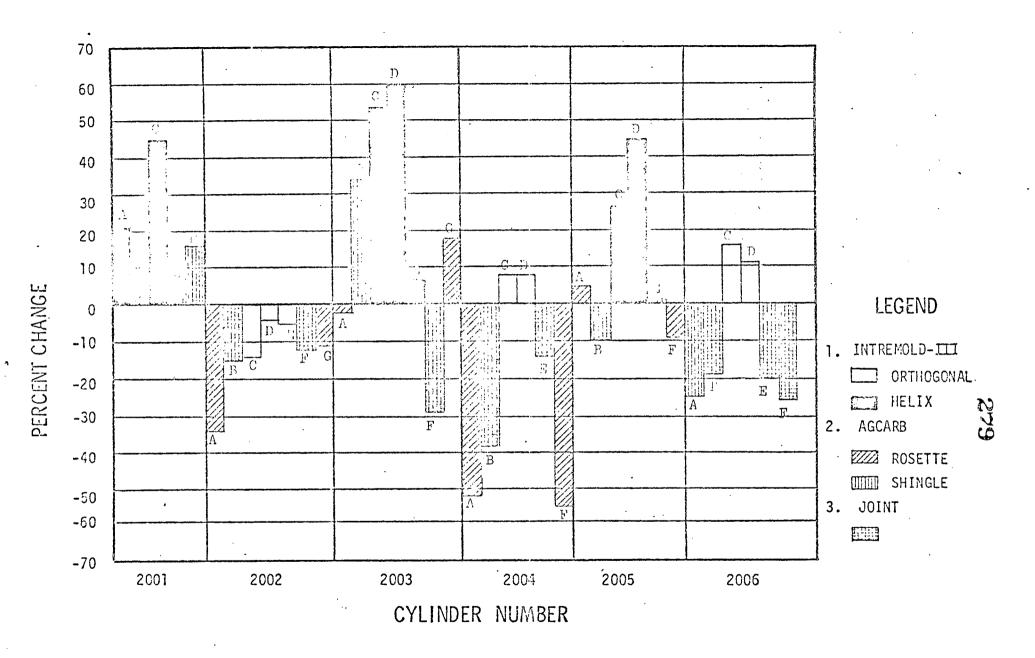


Figure 6. Average Percent Δ 0.D. from Graphitization to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006

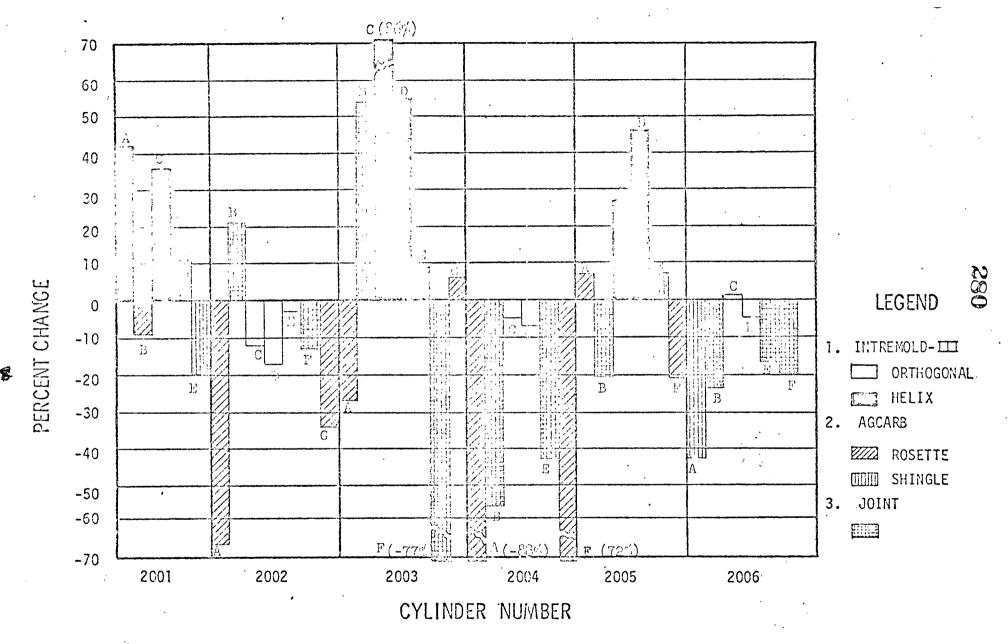


Figure 7. Average Percent Δ O.D. from Original Cure to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006.

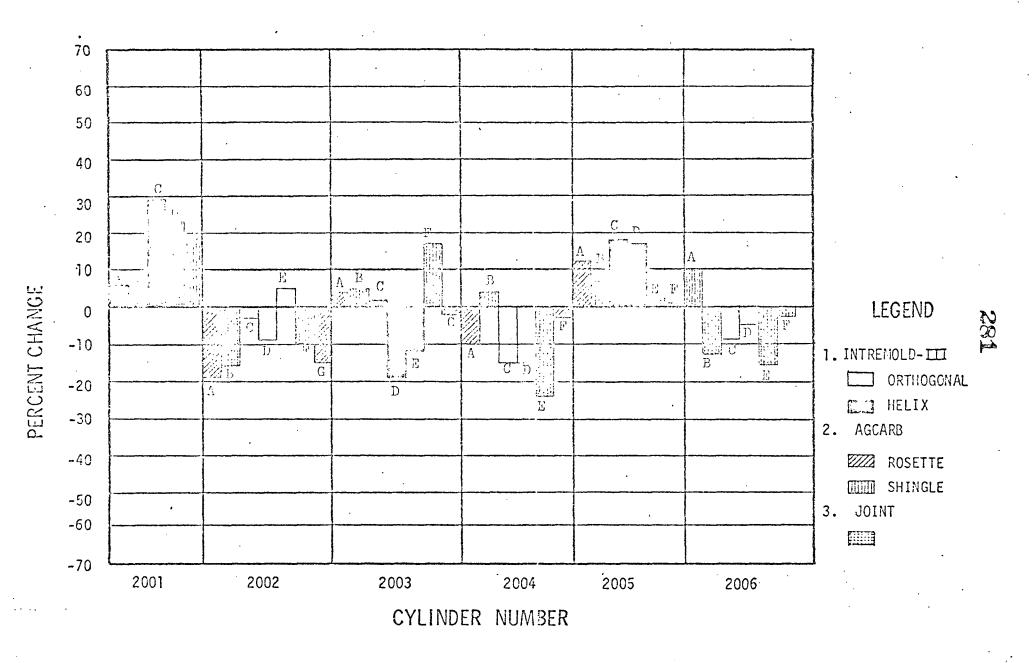


Figure 8. Average Percent Δ I.D. from Graphitization to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006

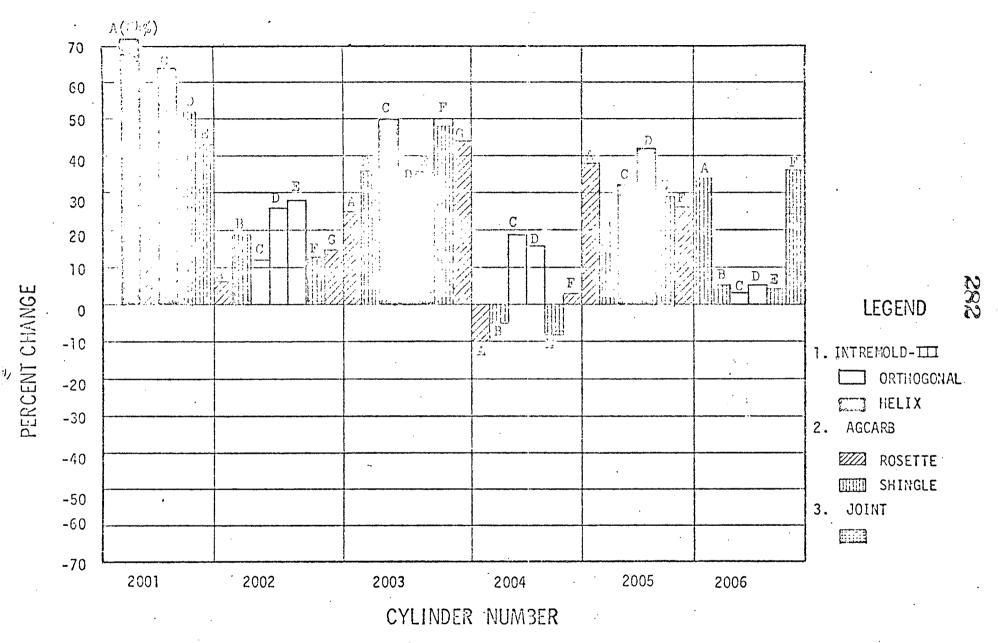


Figure 9. Average Percent Δ I.D. from Original Cure to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006

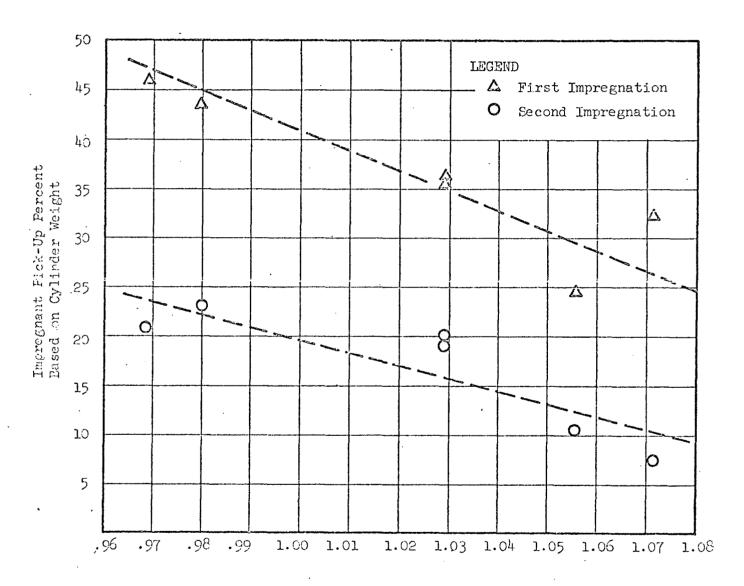
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1. The critical absolute values of correlation coefficient r at a 95% confidence level are ± .811

2. LEGEND

- D Starting specific gravity of cylinder
- E Final specific gravity of cylinder
- F Percent weight pick up first impregnation based on cylinder weight
- H First carbonization specific gravity
- T Second carbonization specific gravity
- W Third carbonization specific gravity
- K Fourth carbonization specific gravity
- Z Fercent weight pick up second impregnation based on cylinder weight .
- R Percent weight pick up third impregnation based on cylinder weight
- V Percent weight pick up fourth impregnation based on cylinder weight.

Figure 10. Correlation Matrix #1 for Specific Gravity and Percent
Weight Pick-up for AGCarb Liner/Intremold III Cylinders N = 6



Initial Specific Gravity (After First Graphitization)

Figure 11. Impregnant Pick-up Percent versus Initial Graphitization Specific Gravity

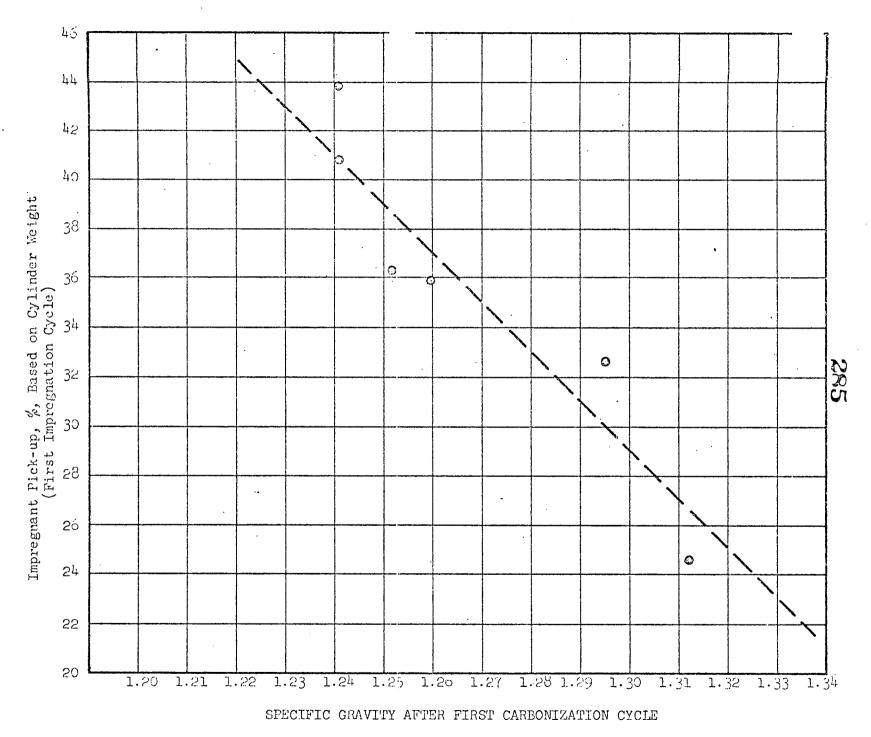


Figure 12. Impregnant Pick-up First Impregnation versus Specific Gravity After First Carbonization.

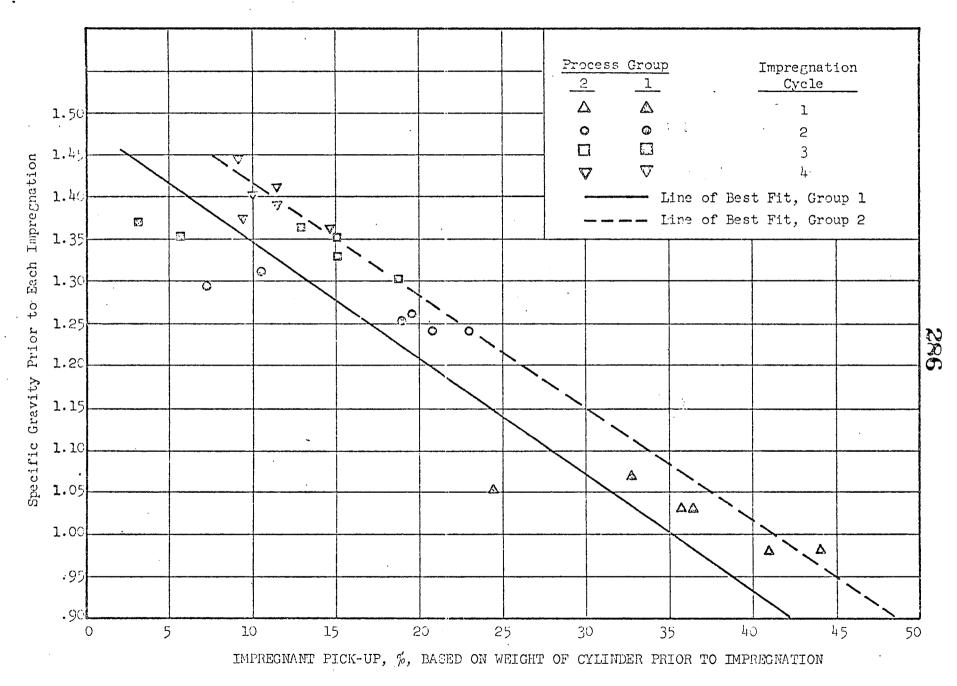


Figure 13. Specific Gravity versus Impregnant Pick-up

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V										

- (1) See Correlation Matrix #3 Figure
 A larger sample size was used for #3.
- 1. The critical absolute values of correlation coefficient r at a 95% confidence level are ± 0.553.

2. LEGEND

- D Viscosity of impregnant prior to impregnation cycle
- E Specific gravity of impregnant prior to impregnation cycle
- F. Temperature of impregnant prior to impregnation cycle
- H Viscosity of impregnant after impregnation cycle
- T Specific gravity of impregnant after impregnation cycle
- W Temperature of impregnant after impregnation cycle
- K Percent volatiles in impregnant-based on weight loss during cure
- Z Percent carbon yield based on weight of cured impregnant
- R Percent pick-up of impregnant based on weight of part prior to impregnation cycle
- V Percent carbon yield based on weight of impregnant

Figure 1^h . Correlation Matrix #2 for Process Variations of 15V/Indene Mixture - N = 13

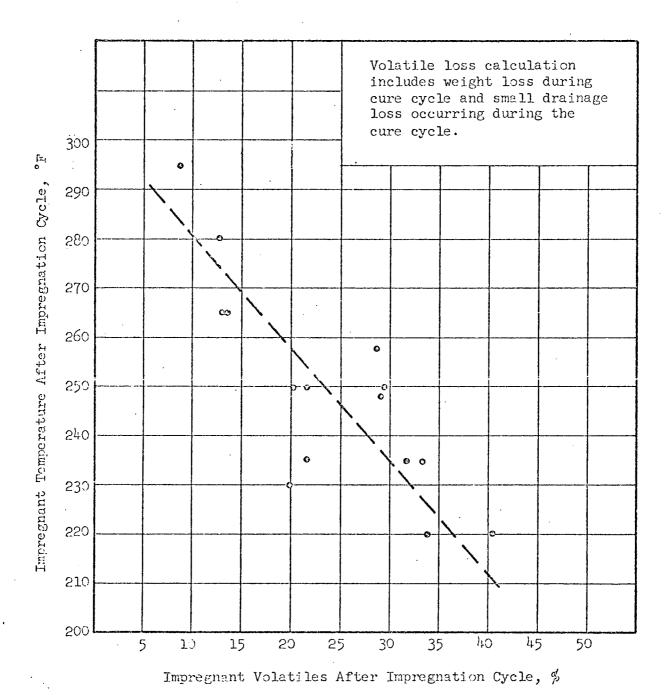


Figure 15. Relation of 15V/Indene Impregnant Percent Volatiles to Temperature at the End of Impregnation Cycle.

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	D	E	F	Н	T	W	K			
D D	-	.727	198	110	.121	012	.137	r della reco ntente		
E		-		234		152				
. F .						065				
Н				-	530		~.77 ⁴			
Т						.112				
ī, ī						-	.015			
K										

1. The critical absolute values of correlation coefficient r at a 95% confidence levelare $^{\pm}$.404

2. LEGEND

- D Viscosity of impregnant prior to impregnation cycle
- E Specific gravity of impregnant prior to impregnation cycle
- F Temperature of impregnant prior to impregnation cycle
- H Percent volatiles in impregnant based on weight loss during cure
- T Percent carbon yield based on weight of cured impregnant
- W Percent pick-up of impregnant based on weight of part prior to impregnation cycle
- K Percent carbon yield based on weight of impregnant.

Figure 16. Correlation Matrix #3 for Process Variations of 15-V/Indene Mixture - N = 24.

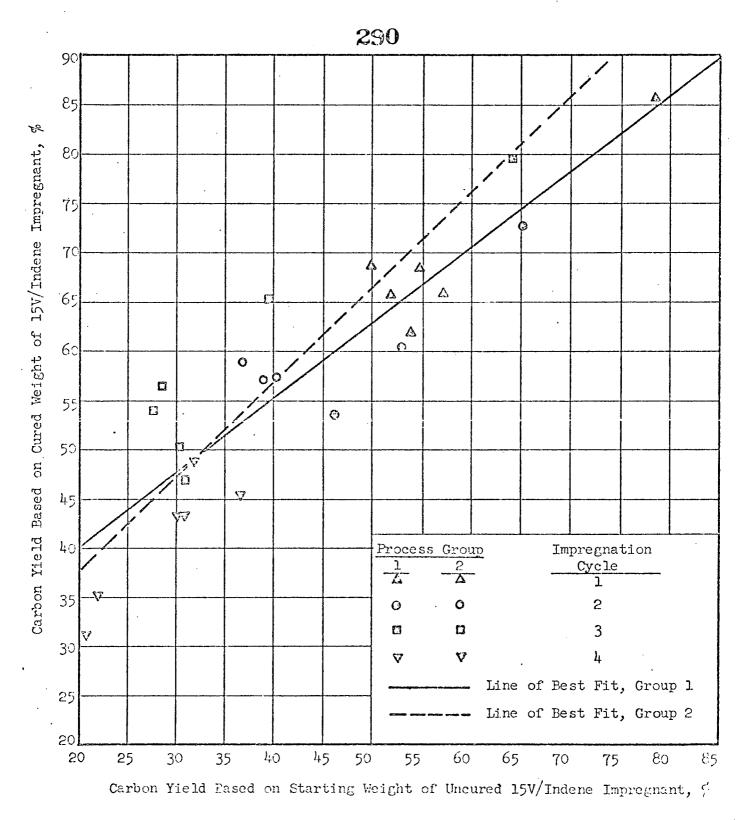
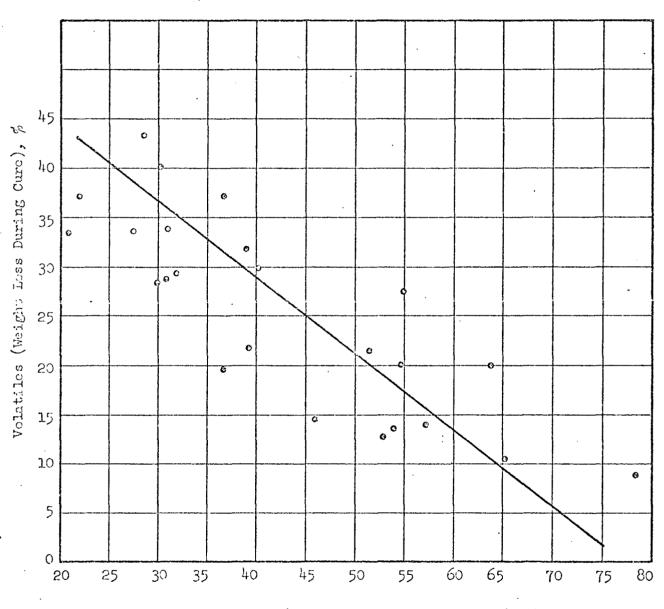


Figure 17. Carbon Yield Percent of Cured Versus Uncured 15V/Indene Impregnant.



Carbon Yield (Based on Impregnant Weight), %

Figure 18. Percent Volatiles Versus Percent Carbon Yield of 15V/Indene Impregnant.

APPENDIX I

INSTRUCTIONS OF SURVEILLANCE PERSONNEL DURING CARBONIZATION AGCarb LINER/INTREMOLD III CYLINDERS

(NOTIFY COGNIZANT ENGINEER IF ANY PROBLEMS OCCUR, DAY OR NIGHT)

- 1. Check temperature and argon flow every hour and initial recorder chart, and record time at each check.
- 2. Maintain argon flow at approximately 10 CFH to 20 CFH at all times; however, the flow rate should be high enough to bubble both the acid and sodium hydroxide solution. Check system for leaks if a flow rate of 30 CFH or higher is required.
- 3. If outlet flow system becomes stopped, change immediately to the second #2 (small outlet and unstop main (1-1/2" tubing) outlet flow. (Record temp. at which stoppage occurred).
- 4. Do not allow any of the tubing to droop. Liquid condensate will fill up the tube and stop flow through the system.
- 5. IMPORTANT. Change cams as needed on controller. This will be every 24 hours. Note: Turn off cam drive switch before changing cams.

 Be sure cam drive switch is back on after cam is changed.
- 6. Check drive arm on controller with T/C readout on circular chart. Variation should never be greater than 50°F.
- 7. Record temperature readout from circular chart on the recorder chart every hour.
- 8. Record argon bottle pressure every hour on recorder chart.
- 9. Change bottles of argon when pressure on gage drops below 200 psi. (The time between changes should be less than 60 seconds.) Switch to a new bottle in the twelve pack. Identify bottle being used and the bottle which has been depleted.

APPENDIX I (cont.)

- 10. As the outgassing starts, the bottles will show gray to green gas being evolved. Record by your initials an (0) if there is outgassing and (N) if there is no visual evidence of outgassing.
- 11. Use a rod to unstop the outlet line in case of a stop-up. (Maintain argon flow, although line may be stopped.)
- 12. Change temperature limiting control to read 100°F above maximum on cam at the time each cam is changed. Temperature limiting control is on strip chart recorder dial right hand top.
- 13. After outgassing has started, turn on tubing heater blankets. Use variac controller to control temperature at the temperature reading on the strip chart. There are two T/C's on the outlet tubing.

 Record the temperatures of the tube T/C's on the strip chart every hour.

NOTE: Do not exceed 1200°F on tapes.

14. There will be condensate in the tygon tubing. If the tube becomes stopped, remove and replace tubing. Save the tube with condensate for analysis.

PART C

INTREMOLD III CYLINDER NONDESTRUCTIVE TESTING

I. INTRODUCTION

The Intremold III cylinders were inspected by X-ray, transmission ultrasonics and penetrants at Aerojet. A study of thermal inspection was performed by the Automation Industries Research Division, at Boulder, Colorado. Some exploratory studies in eddy current inspection were made, and neutron radiography was planned but not carried out at the time work was suspended. Appendix A contains the data reported from the X-radiography, ultrasonic and penetrant inspections. Appendix B is a report on thermal inspection.

II. SUMMARY OF RESULTS

A high quality radiograph shows sufficient detail to identify any probably defect. Without a program of performance testing, destructive analysis and sectioning, it is not possible to determine with certainty what defects are present. Nevertheless, some small flaws are identifiable. Also the structure of the part is shown in detail. It is therefore reasonable to conclude that x-ray inspection would meet the requirements of inspection sensitivity.

Speed and economy are the only questions remaining. Some of the possible flaws would require very precise beam alignment and thus only a small portion of the part could be covered at one exposure. A technique is needed to supplement X-radiography to remedy this defect.

Porosity is the main problem with ultrasonic and penetrant inspection. Unless the materials were made less porous neither inspection would be very helpful. Eddy current inspection does not seem to be worth pursuing at this time. Neutron radiography is indicated only if hydrogenous material is present as a defect, or an un-carbonized adhesive layer needs to be examined more minutely than is possible with X-rays.

If an inspection were to be implemented at this time, the best method would be an X-ray inspection with a technique similar to the one described in this report. Inspection quality would be adequate if enough exposures were made.

The present study deals mainly with materials evaluation and joint construction. It is possible that the overall structural integrity of a finished nozzle extension would present some slightly different problems. Techniques eliminated by material properties, however, would remain unusable. For finished extensions a sonic tap test might be usable and thermal testing would be a candidate.

III. DISCUSSION OF RESULTS

A. X-Radiography

The kind of defects to be expected in intremold cylinders are either discrete low density areas (cracks, voids, porous regions) or inclusions of foreign materials. X-rays are good for detecting such defects. In fact, the density variations present in the intremold cylinders are easily seen on the radiographs. Graphitized resin and the carbon fibers differ sufficiently in density to be distinguished. Of course, cracks, voids and inclusions are conspicuous on radiographs which have enough resolution to show the fibers.

X-rays of 50 and 80 KV energy were used and the tube was fitted with a beryllium window. The film type was R. No screens were used and the film was exposed to the 1.8 H&D to 3.0 H&D range. The focal spot was 0.7 and 2.5 mm. Film to focal spot distances were 41-in. for single wall radial exposures and 48-in. for tangential exposures. The radial single wall exposures were 30 sec, 50 KV, 8 ma.

In general, the pre-impregnated cloth and filament wound portions of the cylinder were relatively uniform density. The radial cell portions showed what appeared as a lack of wetting of the pins. Of course each radiograph shows the cells perpendicularly only on a small part of the film, but

where this view is shown, every cell has a dark outline around the pin. This suggests, in general, that the pins are not wetted by resin, or else absorb enough of it to produce a void at the pin surface during cure and carbonization. Where a large void occurs, it is visible from any angle, but the characteristic lack of wetting at the pins cannot be judged quantitatively except where the pins are exactly parallel to the beam. If such judgment proved necessary, there would be a problem, because the required view is provided over only about 12 sq. in. on the single wall radial exposures. Thus many exposures would be required. This condition, however, appears to be characteristic of the material, and not a defect in the usual sense.

There are more ordinary defects, however. At the bond lines of the joints, voids and delaminations are easily seen. Voids can be seen on the single-wall radial exposures, although their radial position cannot be determined. Delaminations which result from just separation of layers (no material missing) show only on tangential exposures. A tangential exposure shows radial position but covers only a very small area, thus there is a potential problem of time and expense.

The most severe limitation on radiography is the one characteristic of cylindrical objects. That is, some defects can be seen only with tangential exposures, of which many are required for complete coverage.

The usual procedure for covering limitations in radiographic inspection would be to supplement radiography with some technique such as ultrasonic inspection to detect the cylindrical-shell defects. Such an inspection could be used to screen parts prior to radiography. The cylindrical shell defects could be dtected with 100% area coverage and a few tangential radiographs made at cylindrical locations where the nature of the defect would be best shown. In cases of ungraphitized resin, neutron radiography may be logical. The resin would contain hydrogen and thus would show plainly on a neutron radiograph. Any hydrogeneous adhesive would also be conspicuous. Results for neutron radiography are not available, but the ultrasonic inspection was performed.

B. Ultrasonic

It was quickly determined that ultrasound attenuation in the materials was much too high for a pulse-echo inspection. Through transmission is possible at 1 MHz, although results are not especially good. As the radiographs indicate, the parts are quite porous. Where the entire cylinder wall is composed of radial-cell material there is almost no sound penetration. The other regions can be penetrated, but only partial penetration is possible where the materials overlap at joints.

Because of severe attenuation and scattering, as well as the necessity to use fairly low frequencies, beam definition is poor. For this reason, very small defects will not be seen ultrasonically. The radiographs show small defects that ultrasonic inspection will not resolve.

Besides the problems associated with generally poor sound propagation in the radial cell material, it absorbs a great deal of water in an immersion inspection. This would probably cause the material to become more transparent to sound with prolonged immersion. Thus consistent results would require a protective film of latex or similar material.

In general, only gross defects could be seen by ultrasonic inspection. Very large delaminations or possibly large variations in porosity could be detected in some regions. The kind of flaws revealed by the radiographs would not be detected.

Appendix A contains C-scan records of the ultrasonic inspections. These show considerable detail but none of it is really interpretable. The signals were weak, requiring maximum amplifier gain and the fluctuations mapped are rather small. While it might be possible to correlate the indications with some condition of interest, they do not correlate with conditions revealed by radiography. In all probability the C-scan records merely show the normal characteristics of the material.

C. Penetrants

· Isopropyl alcohol was used as the penetrant. The procedure with alcohol is to wet the surface, wipe it and look for residual wetness which will persist around cracks or porosity. The thin film left after wiping quickly evaporates on smooth, non-porous surfaces.

The radial cell material is too porous for penetrant inspection. Tape wrap and filament wound material can be inspected. The penetrant inspection will, however, not be especially sensitive on such materials and will only show cracks open to the surface.

Because of the necessity to use more effective techniques, it is probable that penetrant will prove redundant. It is improbable, for example that penetrants would detect any flaw which would escape x-ray inspection, and a thorough x-ray inspection appears necessary.

D. Thermal Inspection and Eddy Current Inspection

Thermal and eddy current inspection would both yield information more-or-less reflecting heat transfer characteristics. Eddy current inspection is attractive because it is simpler and more economical than thermal inspection with an infrared scanner. Thermal and electrical conductivity are sufficiently related that the electrical conductivity gives a fair indication of thermal conductivity.

In practice, eddy current inspection does not appear very promising. Some success has been achieved with directional probes where carbon-cloth tape was wound to present a single-like ID surface on nozzle cones. Such structures had the fibers in parallel planes when properly wound and the material showed a very directional conductivity. The material was found to vary in conductivity at wrinkles or where there was poor bond between layers. A-3 Polaris nozzles showed these properties.

The intremold materials do not appear to be of the proper structure for eddy current tests. No encouraging results were obtained with any of the cylinder materials. Once more, it appears that x-ray, required particularly for interface defects, will also do an adequate job of revealing any flaws which would be seen by eddy current inspection.

E. Neutron Radiography

Neutron radiography depends upon the fact that thermal neutrons penetrate most materials well, but are most likely to interact with atoms of mass similar to that of the neutron. Thus unlike x-rays, they are absorbed by the lighter elements. Hydrogen-containing materials are much more opaque than most others whereas with x-rays, absorption is almost proportional to density. There are some other elements which are exceptionally good neutron absorbers but they are of no concern here.

Neutron radiography would be needed if there were a problem of incomplete carbonization since it would reveal the presence of hydrocarbons. When hydrogenous resins are carbonized, they lose little weight, but almost all the hydrogen is driven off. Thus x-ray attentuation is only slightly reduced, while the attenuation of a thermal neutron beam is greatly reduced. The presence of hydrogenous material would be conspicuous on a neutron radiograph.

Disadvantages of neutron radiography are mainly expense and inconvenience. A nuclear reactor is the only practical source of the required neutron flux, so parts would have to be shipped to the inspection facility. Also x-ray radiography will give higher resolution. X-rays are obtained from a source spot which can be very small, whereas neutron beams for radiography are obtained by collimating an originally omni-directional thermal neutron flux. A reasonable flux density requires acceptance of less-than-ideal collimation. Where no non-carbon material is present there is little to be gained by neutron radiography.

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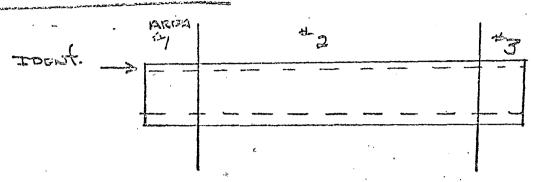
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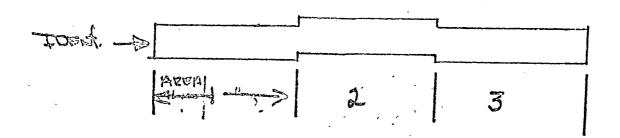
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Appendix A

I.D. SURFACE



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AREA 3 - GOOD

		Appendix A Page ** 1/39309,1139310
ULTRASONIC PROCEDURE AGCS 0731-9	307	PURCHASE ORDER NO.
NAME OF ULTRASONIC LAB.		PREPARED BY
ALRC		D.E. HURST
ADDRESS OF ULTRASONIC LAB.		PHONE NO.
ALRE-SACTO, CAL	LIE	15.3192
NOTE: THIS FORM PROVIDES THE ESSENTIAL INFO		

- 1. Customer approval of ultrasonic procedure to be used.
- 2. Shop instruction for performance of the inspection

3. Report	t record of the p	procedure used.			SPERRY-REFLECTOSECPE					
	SPECIF	FICATIONS		ULTRASONIC INSTRUMENT UM-700						
1. Procedure Control AGC STD. 9014				Make and Model	PULSER/RECEIVER UN.T-IN					
2. Acceptance Standard			·	Recorder (if used)	HELIX RECORDER					
3. Other	3. Other NOT- EVOLUTAL			Make and Mode	el MODEL 319CA					
TRANSDUCER	s(trans)	(REC) 2	3		TEST BLOCK STANDARDS					
Moke	Automati	Gostamotua Gos		Material	NOME					
Туре	15	15		Hole Size	11					
Size	12.25 M	C 1.00 MC		Hole Depths	11					
Rated Frequency				Other	11					
Operated Frequence	ey —			Couplants used	WATER					

ON SKETCH SET UP BELOW, INDICATE:

- 1. Shape of part.
- 2. Directions of scan
- 3. Transduter by number above, for each scan
- 4. Index distance
- 5. Scanning speed

THROUGH TRANSMISSION UTILIZING, 50 INCH COLLIMATOR.
PARTS IMMERSED IN WATER.

DATE

INSPECTION DATA

REFLECTOSCOPE	TRANSHERAPH (C SCAN)
PULSE LEWGTH - MAX. REJECT - 8:30 FREQ - 1.0 GAIN - 1.5 TO FULL	LIPPER LIMITATION—6:30 LOWER 11 - 9:15 INTENSITY — 9:15 NEG.
SIENSHIUHY X10 TRANSDUCERS X10 INCHES	•

NOTE: ADDITIONAL HETRASONIE TECHNIQUE INFO RECONDED ON "C" SCANS.

EDDY CURRENT

Equip- E0-500

PRELIMINARY TESTS DETERMINED PARTIAL CONDUCTIONAL TESTS IF NECESSARY TO BE CONDUCTED.

F C TESTS SUSPENDED

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START OF SCAN

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* Portion of radial cell section

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Appendix A

NOTE: THIS FORM PROVIDES THE ESSENTIAL INFORMATION FOR:

- 1. Customer approval of x-ray procedure to be used.
- 2. Shop instruction for performance of x-ray
- 3. Report record, per MIL-STD-453, of procedure used.

SPECIFICATIONS] FILM	X-RAY EQUIPMENT	RADIOGRAPHIC QUALITY			
PROCESS CONTROL	MAKE	MAKE	PENETRAMETER IMAGE, LEVEL:			
MIL-STD-453	EASTMUM - KODAK	MURELCO, Be TUBE	NONE AVAILABLE			
ACCEPTANCE DEVELOPMENT		KV MAX:	DENSITY, INTERPRETATION ARE			
SEE AHACHED REPOR		150 KV	1.8 to 3.0			
OTHER	SCREENS	FOCAL SPOT SIZE	1.0 10 0.0			
DEVISIOPMENT	NONE	0.7 \$ 2.5 MM				
IDENTIFICATION OF REPAIRS, IF A	NY (R1, R2, ETC.)					

ON SKETCH SET UP BELOW, INDICATE:

1. Direction

2. Distance

Film Positions

11. Film station no's.

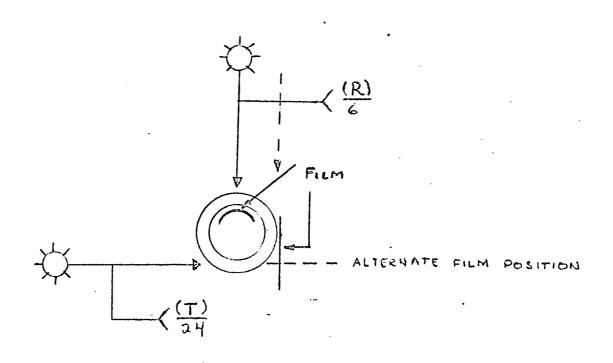
12 Backing (if used)

Time (recommended)
 KV

13. Thickness at time of x-ray

14. Special Instructions

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343 AGCS 0712-1 (REV 8/66) PART NAME PART NO. DASH SERIAL NO. าหลับ" · CYLINDER 11.39309 11-1 VENDOR LAB. S.O./OPER. P.O./DIST. ALRC C11607-015 W.O. NUMBER REFERRAL NO. I.R. NUMBER LEGEND: A. Lack of Penetration K. Shrinkage Porosity 7319-17-113 MATERIAL TYPE 1. Small B. Lack of Fusion L. Shrinkage Sponge SPECIFICATION 2. Moderate -C. Crack . M Shrinkage Cavity 3. Borderline D. Gas Porosity N. Dross CARBON 4. Excessive E. Gas Holes O. Cold Shut HEAT NO. QTY. REC. OTY X-RAYED QTY. ACCER. QTY. REJECTED F. Less Dense Inclusions P. Misrun G. More Dense Inclusions R. Surface Indication MO DAY YR. INTERPRETOR STAMP NO. H. Undercutting S. Film Defect J. Thinning T. Other (Specify) CONTROL NUMBER Acc. Rej. V1 V2 V3 V4 V5 V6 V7 V8 V9 V10 V11 V12 V13 V14 V15 REMARKS OV IL PADIAL PLANE 3447 191 2106 77112 E 01= TRE VISIBLE ON 111 ایت م 12/ 12 11/2.5 7/ Æ., 2/ 43.7 727 122 J. 33. 1.5 0 3/1 120 VALINETS OF MATERIAL 1% 13 221 11 73 72 700 12/12/12/12/12/13 6. 22: 186=795 1/2/2/2 21/1/2 2/11/1/20 Tir. 12 ATT CUTC 11. 11/2/1/2011 X 189500 175 11 134 : _ 1.1 TREA OF PRINT 71 12.10 11 1-- 12. 11/12/1 1111 14 3177260 124 UP FROM END 30000 11/2 1.51 مرتدية 2011 212/19/19/19/19/19/1 OF POSLIBNINATION 10 2120 76 20 6 1.0 4.5.5. 11/11/50 E ARE INDICATIONS 2011 -115 12:17:17 17/22 10 Reproduced from best available copy.

Appendix A Page 12 RAPIOGRAPHIC INSPECTION REPORT 344 AGCS 0712-1 (REV 8/66) DASH C/L SERIAL NO. THRU PART YAME PART NO. 1139310 0-1 S.O./OPER. VENDOR LAB. P.O./DIST. 011608-015 MIRC W.O. NUMBER REFERRAL NO. LEGEND: A. Lack of Penetration K. Shrinkage Porosity 7319-17-113 MATERIAL TYPE 1. Small B. Lack of Fusion L. Shrinkage Sponge SPECIFICATION 2. Moderate C. Crack M Shrinkage Cavity 3. Borderline D. Gas Porosity へのくざのひ 4. Excessive E. Gas Holes O. Cold Shut HEAT NO. QTY. REC. QTY. ACCER. OTY X.RAYED QTY, REJECTED F. Less Dense Inclusions P. Misrun G. More Dense Inclusions R. Surface Indication MO DAY YR. INTERPRETOR STAMP NO. H. Undercutting S. Film Defect J. Thinning T. Other (Specify) V5 V6 V7 V8 V9 V10 V11 V12 V13 V14 V15 CONTROL NUMBER Acc Roj VI V2 V3 REMARKS ∀4 IN STARNED MREA 120 INDICATIONS 30 211 16 11/11/12/10 12:1= 3170VC 31/11/16 122 MATERIAL 11 12 11 -1202 30 3/1/20 10: Reproduced from best available copy. ٤. 1 7

Appendix A 345 RADIOGRAPHIC INSPECTION REPORT Page 13 AGCS 0712-1 (REV 8/66) PART NAME DASH C/L SERIAL NO. PART NO. CYLINDER 11-7. S.O./OPER. 1139564 VENDOR LAB. P.O./DIST. C11609-015 ALRE 17115C W.O. NUMBER REFERRAL NO. LEGEND: A. Lack of Penetration K. Shrinkage Porosity 73/9-17-113 MATERIAL TYPE 1. Small B. Lack of Fusion L. Shrinkage Sponge SPECIFICATION 2. Moderate C. Crack M Shrinkage Cavity 3. Borderline D. Gas Porosity N. Dross CARSON HEAT NO. QTY. REC. RECORD FUL MNORMALIES

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RADIOGRAPHIC INSPECTION REPORT Page 16 348 AGCS 0712-1 (REV 8/66) PART NO. | DASH C/L SERIAL NO. PART NAME VENDOR PRODUCTION 2005 VENDOR LAB. P.O./DIST. S.O./OPER. C11715 -015 ALRO REFERRAL NO. W.O. NUMBER LEGENO: A. Lack of Penetration K. Shrinkage Porosity 7319-17-113 MATERIAL TYPE 1. Small B. Lack of Fusion L. Shrinkage Sponge SPECIFICATION INFO. ONKY
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Appendix A

APPENDIX B

TR 72-18

THERMAL NONDESTRUCTIVE TESTING

OF CARBON/GRAPHITE CYLINDERS

INTRODUCTION

Program Objective

The primary objective of this program was to determine the feasibility and applicability of infrared NDT techniques for evaluating carbon/graphite composite structures. One cylindrical sample, approximately 9.5" dia. x 17" length x 1" wall thickness was submitted as representative of composite structures being investigated by Aerojet Nuclear Systems Co. The sample cylinder contained no known natural or artificial discontinuities. The infrared feasibility studies were conducted to determine if they were capable of detecting any indications which appear abnormal to the apparent normal characteristics of the submitted carbon/graphite cylinder.

Mr. John Amaral of Aerojet Nuclear Systems Co., visited the Boulder Research Center and witnessed the infrared testing on the carbon/graphite cylinder.

The Thermal NDT Technique

The evaluation of many types of materials and laminate structures has proven successful using thermal nondestructive testing techniques. The thermal method of inspection involves the inducement of a heat current within the part and the monitoring of the surface temperature variations. Discontinuities, voids or inclusions within the part alter the normal heat flow and are imaged on the surface as a hot or cool area.

The thermal inspection capability is directly related to the heat conductivity characteristics of the material and by the size and depth of discontinuities being detected. Due to lateral diffusion of the thermal energy, defect detection and resolution capability decreases proportionately with depth of the defect within the material. The thermal NDT applications are therefore best adapted to the inspection of thin materials and to the detection of near surface discontinuities.

TEST PROCEDURES & RESULTS

Front Surface Test (OD Heating and Temperature Monitoring)

This test consisted of heating the sample on the outside surface using two 1000 W tungsten filament bulbs and two 6.5" diameter reflecting and focusing mirrors. The infrared energy was concentrated to a 1" x 1" heating spot on the surface of the cylinder. The heat source is reduced to approximately 124 watts due to filtering used to prohibit stray IR energy from being reflected directly into the camera detector.

The sample was positioned on a rotating fixture to provide uniform rotation for heating and surface temperature monitoring. A variable speed control on the rotator permitted the use of various temperature delay times as the sample was evaluated.

Surface temperature distribution was monitored by a liquid nitrogen cooled infrared pointing radiometer. The surface temperature variations were recorded using an X-Y plot line scan recorder. The Y direction showed location of indications around the circumference of the cylinder and the X direction shows temperature variations.

Temperature monitoring line scans were made circumferentially around the sample at 1" index increments along the cylinder axis. The initial test measurements were made monitoring the OD surface of the cylinder at the trailing edge of the heat source. This technique provided minimum delay between heating and measuring the surface temperature and provides maximum near surface inspection sensitivity. Additional varying depth sensitivity tests were made in selected areas using different delay times ranging from 1/2 second to 24 seconds.

Results

The front surface heating/monitoring technique appeared to show minor variations in surface finish or slightly subsurface material conditions. These variations are shown in line scan recordings 1 thru 9 made at 1" increments across the cylinder. Since the sample contained no known reference standards or natural defects, we were not able to determine true depth of penetration and interpretation of the indications shown.

Additional front surface heating/monitoring results are shown in Scans 10 thru 14. Several scans showed front surface indications which could be correlated with surface scratches, etc. Some indications

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could be altered by slight rubbing of the test surface and were possibly directly due to surface reflectivity.

Thru-Transmission Test (OD Heating/ID Temperature Monitoring)

The second IR technique used to test the sample was similar to the first except the IR camera was positioned to measure the heat that was transmitted thru the sample. This was accomplished by heating the cylinder on the OD surface and using a 45° reflecting mirror positioned on the ID of the cylinder. The mirror reflects the IR energy transmitted thru the cylinder to the camera located near the open end of the cylinder as shown in Figure 1.

Results

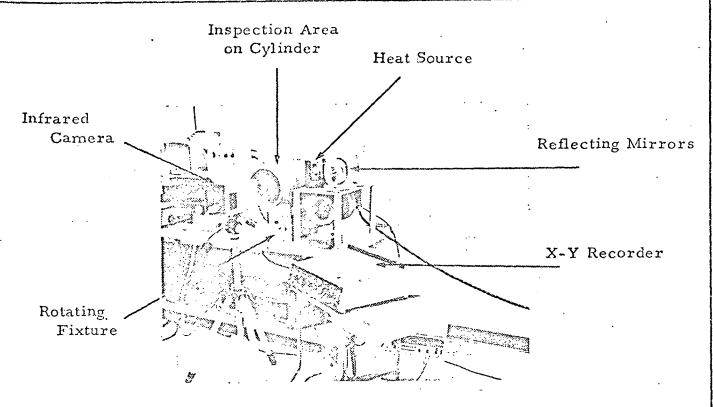
The Thru-Transmission Test (OD heating/ID temperature monitoring) showed a gradual uniform heat buildup on the ID surface of the cylinder. No discontinuity indications were observed. A small piece of masking tape was positioned on the ID surface as a simulated reference indication. The tape showed as a cool spot on the ID surface and is indicated in Scan No. 10.

Thru-Transmission Test (ID Heating/OD Temperature Monitoring)

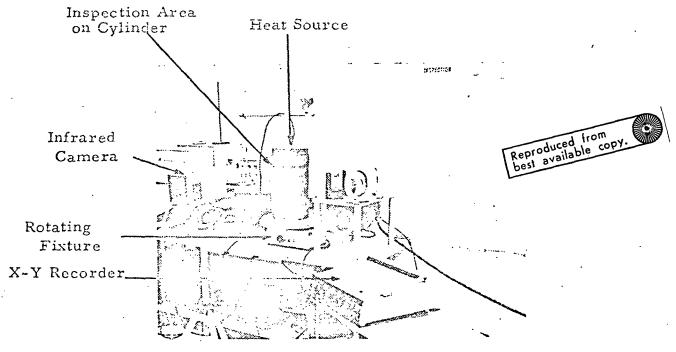
The third technique used was to heat the cylinder on the ID surface using a single 1000 watt bulb and monitoring the temperature increase on the OD surface while the sample was rotating. This test was performed to determine if correlation could be observed between the thrutransmission test and the previous front surface heating/monitoring technique. Delay times up to several minutes were used in the thrutransmission tests.

Results

The ID heating OD monitoring technique showed a gradual uniform OD surface temperature build up circumferentially around the cylinder; however, no changes were noted that appeared to be abnormal to the apparent normal characteristics of the cylinder. There appeared to be no specific correlation with the previous front surface heating/monitoring techniques.



OD Heating ID Temperature Monitoring (Thru Transmission)



ID Heating OD Temperature Monitoring
(Thru Transmission)

Figure 1. Typical Infrared Inspection Setup for Evaluating Carbon/Graphite Cylinder, P/N 1139583

CONCLUSIONS AND RECOMMENDATIONS

The infrared thermal studies described above have been limited to one sample and were performed without the availability of calibration reference standards. The information obtained is therefore quantatively limited and is relative to one test sample. However, the following conclusions may be considered:

- 1. The thermal tests showed some temperature variations which appeared to be associated with the front surface finish or possible subsurface material conditions.
- 2. Thermal energy was quite easily and uniformly transmitted thru the cylinder wall from both the ID or OD.
- 3. There appeared to be no significant correlation between the thru-transmission tests and the OD surface heating/monitoring technique. This was possibly due to heat diffusion caused by the lengthy delay periods required for heat penetration thru the cylinder wall.
- 4. Some visual surface scratches showed as significant indications while heating and monitoring from the OD surface. This would indicate the possibility of thermally detecting surface or slightly subsurface cracks should they occur in a similar structure. However, we observed that some surface indications could be easily altered by slight rubbing or erasing and were possibly caused by direct changes in surface reflectivity.
- 5. The relatively uniform heat flow thru the cylinder wall would also indicate the possible feasibility of using thermal techniques for detecting laminations or defects contained within the structure wall. Since no known defects or reference standards were available, we could not establish defect size detection capability.
- 6. The infrared thermal techniques were relatively easy to apply to the sample cylinder and could possibly lend themselves to inspection of larger scale production parts.

Recommendations

The following recommendations should be considered for completion of this investigation and possible follow-on work.

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- 1. The sample cylinder should be destructively analized and the results correlated with the thermal scan lines to determine the significance of the recorded indications. This correlation should be conducted specifically with the results shown on Scans No. 1, 3, 4, and 7.
- 2. Some correlation analysis should also be made of the surface scratch indications to determine if they are in fact only surface scratches and to determine if they have significant depth.
- 3. Additional thermal evaluations should be performed on similar graphite/fabric samples containing known reference notches, test holes, and simulated laminations. Use of reference standards will permit the selection of proper heat/scan delay times and prove the capability and limitations of the thermal inspection techniques as applicable to carbon/graphite structures.

Note: The original line scan recordings of the thermal inspection results were submitted directly to Mr. J. Amaral of Aerojet Nuclear Systems Co. They will be duplicated by Aerojet Nuclear and added to this report.

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